METALLURGIA

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(INCORPORATING THE METALLURGICAL ENGINEER)

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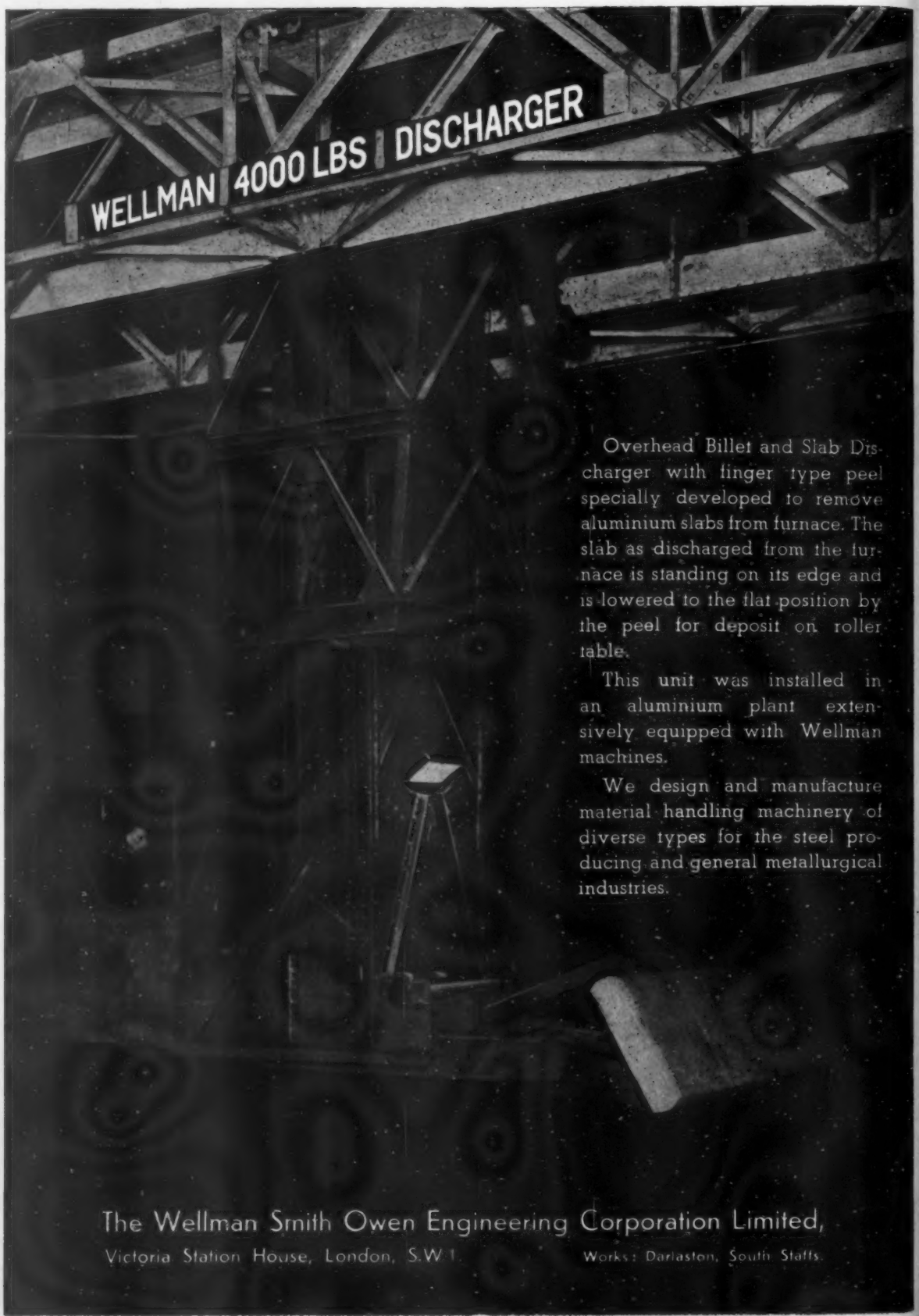

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Flame-Cleaning Steel Structures

With Apparatus Simple and Easy to Operate

IN the preservation of iron or steel surfaces against corrosion, the preliminary treatment prior to painting, in order to thoroughly clean the surface and leave it also dry and warm, is now well known to be as important as the painting itself. A primary factor or result in such prior treatment and a measure of its efficiency is increased adhesion and durability of the paint coating. Research into the different methods of pre-treatment of metals, notably iron and steel, has in recent years and especially under the stress of war demands been carried out on a large scale and results of considerable chemical and physical interest have accumulated, and further enlarged the already extensive literature of corrosion and its prevention. Two of the most effective methods of preparing iron and steel surfaces are phosphating and flame priming or cleaning, of which the latter will be briefly considered here. It is now used on a large scale both in this country and the U.S.A. The method most commonly employed is that of oxy-acetylene flame priming, by which, according to an estimate by Anderson in a recent paper before the Engineering Institute of Canada, some 20,000,000 sq. ft. of steel surface are treated per month in Canada and the U.S.A.

In the fifth report of the Corrosion Committee of the Iron and Steel Inst., 1938, it is stated that, by heating weathered steel before wire brushing and painting, longer paint life was obtained through the heating whereby the surface was thoroughly dry and warm immediately prior to the application of paint; for it has been found that as little as a single molecular layer of moisture or other matter on the surface will prevent satisfactory bonding of the base coat and allow corrosion to continue after painting. On a warm, dry and thoroughly clean surface the paint will flow more readily and adhere more strongly. The value of proper cleaning beforehand is again emphasised in B.S. Specn. 1160/1943; and, it may be added, also figures prominently in the extensive patent and general literature of phosphating and other metal surfacing processes.

Flame cleaning, or priming as it is more usually termed in the U.S.A., is defined as a method for intimately scrubbing the surface with high-velocity high-temperature oxy-acetylene flames, in order to remove all loose scale and other contaminants and leave the surface entirely clean, warm and dry. Both the physically absorbed or occluded and also the chemically-bound water is driven off, leaving only stable oxides, and all high temperature or annealing scale removed due to differential expansion between the scale and base metal or within the scale itself. In addition other contaminants such as oil and acid salts are consumed or disintegrated by the flames, and may be the more readily and completely removed together with the loosened scale, etc., by wire brushing.

The priming coat of paint should be applied immediately whilst the metal surface is still warm (100°–120° F.) and before recondensation can take place. The surface is thus thoroughly protected and inactive; cleaning, painting, and setting times are reduced; and painting can be done under cold or damp conditions, thus further economising time, a point of great importance in continuous work. With new structural steel and plate the shop or priming coat is in this way well preserved and not disturbed by mill scale loosening or flaking, a frequent cause of incipient corrosion even under the finishing paint coat. Similarly in the re-conditioning of old steel structures it is usually necessary, or at least very desirable, to remove the old paint, down to the priming coat or better still to the bare metal, together with all loose scale, rust and contaminants of all kinds. This is most effectively accomplished by the oxy-acetylene flame-cleaning process.

Any contaminant on the surface, including moisture, will prevent effective bonding of the paint, and the slightest trace of water will cause rust formation under the paint. The moisture may be present in rust or other material as physically absorbed or chemically combined and both forms must be completely removed so that the surface of the metal is thoroughly dehydrated, involving the fast application of a fairly high degree of heating. Essentials of such heat application are: (*a*) Maximum temperature possible to give instantaneous conduction of heat through scale layers, etc., to metal surface; (*b*) a dry flame so that no moisture as a product of combustion is deposited; (*c*) a clean flame, yielding no smudge, soot or other undesirable combustion product; (*d*) a flexible flame which may be readily adjusted to a required contour or spread out over a wide area; (*e*) a speedy flame so intense as to permit fast action over a maximum area in a given time.

Comparative tests have shown that the oxy-acetylene flame fulfils these various requirements more effectively than any other—either the oxy-hydrogen or the oxy-propane. Both these latter suffer under the disadvantage of having a lower flame temperature and of being less clean and moisture-free in operation.

During the war the use of flame-priming iron and steel surfaces was extensively used, and special conditions of use have been drawn up, for example, by the U.S. Navy in their painting specifications. This was emphasised by Anderson, in the paper read at Toronto, already referred to, in which he says that numerous and exhaustive tests have been made by the U.S. Navy and by the American Institute of Steel Construction, both in ordinary inland industrial atmospheres and in marine atmospheres on board ship, using different types of first coat paints and comparing flame

priming with other preparatory methods such as sand-blasting, hand-brushing, etc., but not apparently with chemical baths or the like treatment.

The same author refers to the reducing action of the flame gases in addition to their dehydrating effect and formation of anhydrous oxide. This latter by reduction is converted to ferroso-ferric oxide or black magnetite indicating that a very stable surface has been obtained. Further reducing action converts the magnetite to sponge iron if the flames are moved more slowly, but such further action is not necessary, nor indeed desirable. The loose powder can be removed by light brushing, and this should be done whilst the surface is still warm and immediately prior to painting.

Another important point stressed by Anderson is to have a maximum amount of tight or firmly bonded scale intact on the steel surface and to paint it before rusting can spread from an exposed area to undermine the protective layer. Although, as he says, this ideal is hardly possible in actual practice flame-priming probably provides the nearest artificial approach to such a condition. In the U.S. Navy specifications it is stipulated that mill scale be left intact as far as possible by insuring that there shall be a minimum of rusting before flame-priming can be applied. It should be added that flame-priming is said not to remove the tightly held corrosion-resistant scale but only the loose variety. This, however, is not altogether clear or quite consistent with the alleged loosening effect of flame-priming on the scale due to a two-fold differential expansion action. In any case it would seem that the extent to which the so-called tightly held scale is affected by flame-priming—*i.e.*, converted into the loose removable type depends on the temperature of the flame and the rate at which the flame jets are moved over the surface.

In addition to rust exposed surfaces very often have also a mixture of acid salts which are for the most part hygroscopic and attract and hold moisture. Wire brushing only is not usually very effective in their removal, but flame-priming decomposes and dehydrates these contaminants and makes them much more easily removable.

The apparatus required is very simple and easily operated. It consists essentially of flame-priming heads or series of jets 1–12 in. wide which can be readily attached to standard welding blow-pipes. Two different types of nozzle are available, flat or round. The former is used for cleaning flat surfaces free from projections, whilst the round is used for all riveted and bolted areas, corners, recesses, etc., where a flat nozzle is not so effective. The flame-cleaning nozzle, attached to an extension piece, is fitted to a special type oxy-acetylene high-pressure blow-pipe, which in turn is connected by lengths of rubber tubing to the oxygen and acetylene supply. There are two types of flame-cleaning equipment: one type for light-duty work, for use with the 2–4 in. flat nozzles and the round; a second type for heavy duty with 2–12 in. flat, and the round.

British Standard Marking System for Grinding Wheels

THE manufacturing members of the Abrasive Industries' Association have, for some time, given special attention to the question of uniform practice for the marking of grinding wheels. The need for some standardised system of marking between one manufacturer and another has long been felt. Systems have been introduced in the past, mainly abroad, but they have not been satisfactory in use. Recently, however, a system has been adopted by the Grinding Wheel Manufacturers' Association of America, which the Abrasive Industries' Association considers the most feasible so far developed.

The system uses alphabetical letters to denote hardness in all types of bond and the range of grades runs from the softest at the beginning of the alphabet to the hardest at the end, this being the method hitherto in use by the majority of grinding wheel manufacturers. It is, however, emphasised that wheels of similar marking, made by different manufacturers, will not necessarily give the same grinding action. This is due to the impossibility of measuring the physical properties of bonded abrasive products in terms of their grinding action in use. Thus, the system is one of standardised markings only, and it applies to grinding wheels and most abrasive products of whatever size, type, and shape, but it does not apply to diamond wheels.

The marking consists of four main parts—abrasive, grain size, grade, type of bond. In addition to these four markings, the manufacturer may include any or all of the following. A prefix denoting the manufacturer's symbol for the exact type of abrasive used in the wheel; a structure symbol which can be inserted immediately after the grade mark and prior to the latter denoting the type of bond; a suffix denoting the manufacturer's symbol for the exact type of bond or other identification.

The main types of abrasives are aluminium oxide and silicon carbide which are symbolised by the letters A and C respectively. In cases where variations in any of these two types are used by a manufacturer, a prefix may be used according to the choice of the individual manufacturer.

The grain sizes are indicated by numbers, which refer to the number of meshes per linear inch through which the grains have passed. Thus 8 to 24 inclusive are coarse, 30 to 60 medium, 80 to 180 fine, and 220 to 600 very fine grain size. In cases where the wheel maker wishes to indicate a special grain combination, this can either be denoted by a symbol after the grain size numerals or by the use of two grain size numbers.

The grade or hardness of the wheel, for all types of bonds, is indicated by letters of the alphabet ranging from A to Z, soft to hard. The main range of grinding wheels commonly in use falls within the range F to T.

The structure of the wheel, which is governed by the spacing of the grains, is indicated by a number. Numbers 1 to 15 cover the range in general use, and these indicate the grain spacing. Structure 1 is extremely dense, and the higher the number the more open the structure.

The type of bond used to hold the abrasive is indicated by letters, as follows: V for vitrified, B for resinoid (synthetic resin), R for rubber, E for shellac, and S for silicate bond.

The suffix, which may be used at the discretion of the manufacturer, may take the form of letters, numerals, or both. It is provided so that any special identification symbol used by the manufacturer may be incorporated in the marking.

Mineral Constituents of Arc-Welding Slags

By R. C. Vickery, M.Sc., Ph.D.

A study is made of the reactions at high temperatures of the major constituents of arc-welding electrode coatings FeO, MnO, TiO_2, and SiO_2. Experimental procedures were such as to closely approach the conditions of slag formation during welding, and the samples of slag thus obtained were examined petrographically. A sequence is suggested for the interaction of the electrode coating constituents, and observations are made on the practical applications of the findings.

IT was initially intended to investigate the characteristics of slags produced by various electrodes in arc-welding, numerous difficulties arose, however, mainly connected with the obstacles which presented themselves during the standardisation of the conditions in force during slag formation in welding. The fact of glass formation could be overcome, but many other factors such as current variation, arc length, etc., were nearly impossible to control. Attention was then turned to the identification of the coating constituents—the results of the author's investigations on this subject have already been published[1]; it was, nevertheless, felt that an investigation of the slag constituents would yield extremely interesting results, and a modification of the original programme was adopted in which, instead of examining slags from different electrodes, the mineral reactions produced in one type of electrode coating were referred back from the slag to the original constituents via intermediate melts. A fuller description of the process of investigation is given in a later section.

Geochemists and ceramists have long been in possession of information on mineral reactions which welding research investigators are only just beginning to apply. Even so, this available information is incomplete for whereas ceramists employ temperatures reaching normally only as far as ca. 1,500° C., the temperature of the welding arc has been estimated at 3,000° C.

A little information is available via mineralogists on mineral reactions at high temperatures, but such is rarely applied by the investigators to welding operations, whereas in the former case equilibrium is usually established, in welding it is rare that the constituents of the slag can be resolved. As the following notes will show, it may be considered possible to extrapolate experimentally obtained data, and thus gain an insight to the reactions between the mineral constituents forming the slag.

The constituents of welding electrode coatings are multitudinous, and even more so are the combinations between them; it does not require much investigation however, to ascertain that probably the most generally employed materials, and those which usually constitute some 80–90% of ferrous electrode coatings, are, silica, titanium, manganese and iron as compounds. The ternary system FeO-MnO-SiO_2 has been variously investigated by Whitely and Hallimond[2], Benedicks[3] and Maddocks[4]; of these, the latter has probably made the most comprehensive investigation. We come, however, to the effect of the inclusion of titanium, but, unfortunately, no really specific investigations of either binary, ternary, or quaternary systems of TiO_2 with the other oxides have been made. The work of Smolensky[5] on $MnSiO_3$-$MnTiO_3$ is an exception, but even this appears incomplete. Several instances have been given of the mineral constituents of either acid or open-hearth slags containing titania in small amounts, but even in this respect the author has been unable to find reports of a comprehensive study of the subject. The classic compilation of phase diagrams by Hall and Insley[6] forms the basis for most excursions in the subject.

Experimental Procedures

Under the conditions and amenities existing at the time the work here recorded was carried out, it was not found possible to rectify the faults indicated supra relating to arc length, current stabilisation, etc., and the effects of their variation, indeed it was not considered that research on such a subject would come within the scope of this initial investigation.

On consideration of the main constituents which would form the main body of a mild steel electrode coating of the more usual type, a coating was developed which would enable the electrode to be deposited in the normal way by a welder of average experience—viz.:

SiO_2 : 10%
TiO_2 : 60%
MnO : 20%
FeO : 10%

The silica employed was the purest precipitated, ignited material available; titanium dioxide was of the purest white grade obtainable—98–99%, and the ferrous and manganous oxides were prepared as by Maddocks, by the thermal decomposition of their oxalates under *vacuo*. The whole mass, after thorough dry mixing *in vacuo*, and passage through a 100-mesh sieve, was mixed to a fluid mass by the addition of 2% sodium silicate in water, and coated on to 8 S.W.G. mild-steel wires to a thickness of 6·25 mm. *i.e.*, the coating thickness was ca. 1·2 mm. The electrodes thus prepared, after drying at 110° C., and baking at 200° C. *in vacuo* in both cases, were employed for depositing metal on to ordinary mild steel at a current of 140 amps. D.C., the electrode being attached to the negative pole of the current generator. After permitting the weld to cool at the normal rate, and taking precautions to avoid loss, the slag was removed from the surface of the weld metal by light chipping with a small geological hammer. The pieces of slag thus obtained were bulked together, and, except for a few larger pieces retained for separate examination, reduced to a powder capable of passing a 30-mesh sieve, and submitted to the series of experiments A, detailed in the following section. The larger

1 *Metallurgia*. **31**. 119. 1945.
2 *J. Iron and Steel Inst.* No. 1. 199. 1919.
3 *Non-Metallic Inclusions in Iron and Steel*. London. 1930.
4 Iron and Steel Inst. Carnegie Schol. Mem. **24**. 51. 1935.
5 *Z. Anorg. Chem.* **73**. 299. 1912.
6 *J. Amer. Ceram. Soc.* **16**. 456. 1933.

portions of slag were treated as in B, of the same section.

The method and apparatus employed for the experiments were modifications of those used by Shepherd *et al*[7]. The design of the furnace employed however differed, and is shown in Fig. 1. A tungsten resistance type was used. In order to attain the higher temperatures necessary, it was essential that the furnace should exhibit a small concentrated heating area—this combined

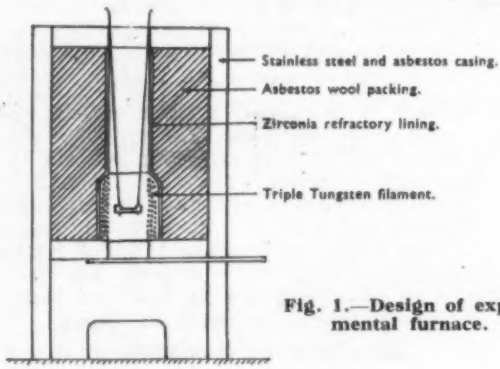

Fig. 1.—Design of experimental furnace.

with the consequent reduction in size of the whole assembly made the apparatus very compact and convenient. In experiments up to 1,500° C., samples of the material under investigation were enclosed in a small piece of platinum foil, and suspended in the furnace by platinum wires at temperatures over 1,500° C., small plates of zirconia were excavated into small crucibles to contain the sample and were suspended in the furnace by tungsten wires.

The temperature attained in the furnace was regulated by a sensitive resistance previously calibrated against a Pt/Pt.Rh thermocouple up to 1,500° C., and against a W/Mo thermocouple in an atmosphere of hydrogen up to 2,500° C.

In order to simulate the conditions in force during the welding operation, the general procedure adopted was to insert the sample and container into the furnace already at the requisite temperature, allow sufficient time for the sample to attain that temperature (four to five minutes was found to be the average period required), and then by releasing the suspension wires, the container and sample were dropped into and quenched by, a silica trough of mercury. The sample thus treated was recovered and examined microscopically for changes in crystalline phases and mineral identity on the basis of the procedure suggested by Tenenbaum and Joseph[8] for examination by reflected light. The actual treatment of samples varied slightly, as will be indicated in the following sections.

Three series—A, B and C—of experiments were carried out. Series A consisted of examination of treated samples of crushed slag; B of large uncrushed slag particles; and C of the original mineral mixture as removed from rods after the drying process. In connection with series C, a comparison was made between the mineral constituents of the prepared electrode coating and the original dry mixture. Owing to the precautions adopted in mixing and drying of employing vacuum apparatus, only slight evidence of oxidation was found in the ex-electrode material, although in the ferrous oxide particles, two outside layers appeared to have been formed; an outer very thin layer—ca. 5μ thick—of riebeckite ($2NaFe(SiO_3)_2\ FeSiO_3$), and an inner layer—ca 10μ thick—of viridite ($4FeO.2SiO_2 \cdot 3H_2O$) surrounding the inner core of unchanged ferrous oxide. The manganous oxide particles were also slightly modified externally to braunite ($3Mn_2O_3 \cdot MnSiO_3$). The dry unincorporated samples showed great oxidation of both manganous and ferrous particles. The rapidity of reaction of the silicate solution, however astonishing at first sight, is, in the light of Bliss's work[9] on the reactions of ferro-manganese with silicate solutions, only to be expected; what is shown, however, is the further possibility of mineral silicate formation in the geological field by infiltration of silica-bearing solutions into unoxidised iron and manganese beds.

The gross oxidation of the dry, unincorporated iron and manganese oxides was only as was anticipated in view of their great tendency to oxidise in air.

Experimental Results

Series A: The crushed slag, after well mixing, was subjected to temperatures varying from 2,500° C. down to 850° C. The procedure adopted was that described supra—insertion of the sample in the already heated furnace, and after attainment of the requisite temperature, quenching in mercury and examining microscopically. Table I records the results obtained in this series.

TABLE I.

Temperature of Quenching. °C.	Phases Observed.
2,500	Glass.
2,000	Glass ilmenite.
1,750	Ilmenite, manganofayalite in glassy matrix.
1,500	Ilmenite, fayalite with trace alleghanyite in glassy matrix.
1,250	Glass—unchanged.
1,000	Glass—unchanged.

The fact that no change in structure was observed below 1,500° C., is quite in keeping with the deductions from classical phase diagrams, even though the inclusion of titanium is known to decrease the viscosity of such slags as those investigated here by depressing the melting point. It may be considered that the primary crystallisation of the ilmenite removed a sufficiently large quantity of titanium from the active field to accelerate the cooling rate so that initially the manganofayalite crystallised out which, at lower temperatures resolved itself into its probable constituents alleghanyite ($5MnO.\ 2SiO_2$) and fayalite (Fe_2SiO_4).

Series B: In this sequence small, compact pieces of slag about 0·25 cc. in volume were subjected to the treatment applied in series A to the crushed slag. The reason for this apparent duplication was that the larger pieces would lend themselves more readily to photographic reproduction, and further the process of mineral degradations, etc. would be more easily observable in such a bulk. On the basis of the previous series a slightly different and more comprehensive temperature sequence was employed. The results obtained in this series of experiments are recorded in Table II.

It is noteworthy that in neither series A nor B was uncombined quartz found. The primary crystallisation of ilmenite is once again exemplified, whilst the transitory appearance of magnetite before its apparent conversion

7 *Am. J. Sci.* 4th Series. **28**. 293. 1909.
8 *Blast Furnace Steel Plant.* **29**. 403 *et seq.* 1941.
9 *Footprints.* **7**. June, 1934.

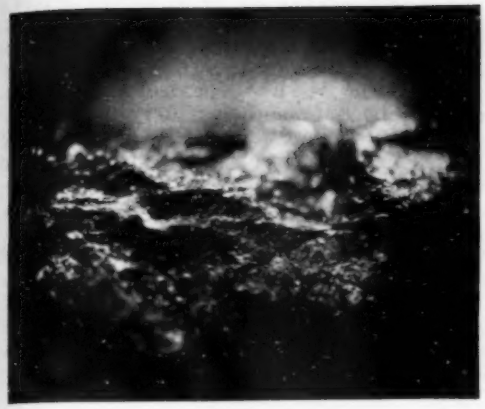

Fig. 2.—Manganofayalite. × 600.

to manganofayalite would appear to support the theory of discontinuous reaction principle as first enunciated by Bowen[10]. Further evidence is given of this by the appearance at 1,650° C. of alleghanyite and tephroite

TABLE II.

Temperature of Quenching. °C.	Phases Observed.
2,500	Glass with minute central ilmenite.
2,250	Glass + ilmenite and magnetite.
2,000	Glass + ilmenite + manganofayalite and trace magnetite.
1,850	Glass + ilmenite + manganofayalite and fayalite.
1,650	Ilmenite + fayalite + alleghanyite–tephroite transformation in glassy matrix.
1,500	Ilmenite + fayalite + tephroite with alleghanyite in glassy matrix.
1,400	Ilmenite + fayalite + tephroite with trace acmite in glassy matrix.
1,200	Glass unchanged.

(Mn_2SiO_4) in place of the manganofayalite. Fig. 2 illustrates the initial manganofayalite, whilst Fig. 3 shows the alleghanyite–tephroite transition; some remnants of manganofayalite can be discerned in the darker alleghanyite. Completion of the transition is apparent at ca. 1,500° C. The appearance of acmite at the lower temperatures can only be explained on the basis of alkalies present in the waterglass employed as binding agent; it must be remembered that on heating acmite ($NaFe(SiO_3)_2$), or rather on attempting to prepare the mineral by melting together the component oxides, one usually obtains magnetite in a glassy matrix, it would appear, therefore, once again that the discontinuous reaction principle holds good in the case of welding slags. The minute inclusions of acmite may be observed in Fig. 4.

Series C: In this sequence the roughly-comminuted coating material removed from the experimental electrodes was subjected to high temperatures in the same way as the previously-examined slags. In this case, however, a rising temperature scale was employed, and particular attention was paid to the observation of minute inclusions. Table III records the results observed. As previously indicated, riebeckite, viridite and braunite were formed in the initial mixture, but

TABLE III.

Temperature of Quenching. °C.	Phases Observed.
600	Hematite + magnetite + quartz + titania + polianite + acmite + traces fayalite and riebeckite.
750	Hematite + less magnetite + quartz + titania + hausmannite + traces riebeckite.
850	Hematite + quartz + fayalite + titania + hausmannite + braunite + astrophyllite.
1,000	Less hematite + fayalite + quartz + ilmenite + titania + braunite.
1,200	Fayalite + ilmenite + quartz + braunite with tephroite and narsarsukite in glassy matrix.
1,400	Fayalite + ilmenite + tephroite with alleghanyite in glassy matrix.
1,500	Fayalite + ilmenite + tephroitic alleghanyitie in glass.
1,650	Fayalite + ilmenite + alleghanyite in glass.
1,850	Manganofayalite + ilmenite in glass.
2,000	Traces magnetite and little ilmenite in glass.
2,250	Traces ilmenite in glass.
2,500	Glass.

these were rapidly decomposed. The low temperature transformation of braunite is rather peculiar, and can only be attributed to the possible reducing action of acmite with the consequent production of polianite (MnO_2). In this connection, and at the temperature employed, the production of polianite in preference to pyrolusite is only to be expected, in view of the normal water content of the latter. The disappearance of acmite with oxidation of the polianite to hausmannite (Mn_3O_4) and magnetite to hematite would appear to afford further evidence for the reducing activity of acmite. The first appearance of fayalite at 850° C. seems to indicate the lowest temperature for its formation under the conditions existing, whilst its quantitative increase combined with the momentary appearance of

10 *J. Geol.* **30**. 177. 1922.

Fig. 3.—Alleghanyite-tephroite transition. × 600.

Fig. 4.—Acmite inclusions. × 1,200.

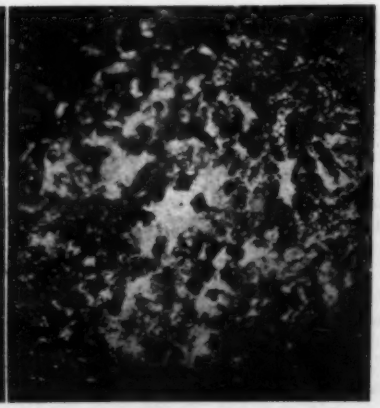

Fig. 6.—Fayalite, ilmenite and alleghanyite in glass. × 800. Specimen at 1,650° C.

These illustrations slightly reduced on reproduction.

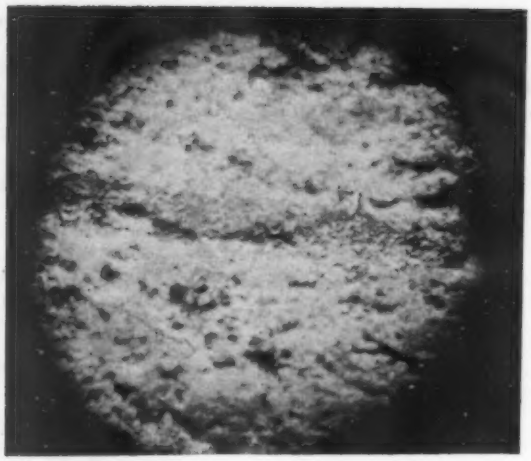

Fig. 5.—Band inclusions of narsarsukite in tephroite and fayalite. × 1,200.

astrophyllite and steady decrease in the hematite content appear synonymous. The finding of narsarsukite at 1,200° C. at the same time as the initial appearance of glass is peculiar, and must be regarded as exceptional, indeed, the two phases were found in quite separate localities in the specimen examined; as shown in Fig. 5, the narsarsukite appeared as a narrow band inclusion in a ground mass of tephroite and fayalite. Tephroite itself was apparently the transition phase between braunite and alleghanyite, which might only be expected. The phases observed at 1,650° C. in a small area of the specimen are shown in Fig. 6. From this point the transformations and sequelae are normal and elementary; combination of the alleghanyite with the fayalite gave manganofayalite, which finally decomposed, leaving traces of magnetite. Ilmenite as one of the primary crystallising constituents of a magma is seen as far as 2,250° C., but even this remains in the glass at higher temperatures.

Discussion of Results

From the experimental evidence it is possible to outline a probable sequence of mineral reaction and transformation, as shown in the diagram Fig. 7. The indications of reversibility of some of the reactions at the higher temperatures are based on the phases observed in the re-treated slag specimens. The diagram should need little explanation, but two interesting points arise which must necessitate comment. It is generally accepted that the formation of amphiboles, such as riebeckite require the presence of a volatile flux for their formation [11]; in the initial mixture of the components, care was taken to ensure the absence of any extraneous catalytic material, it would seem, therefore, that water itself can provide sufficient incentive for such reactions and that the formation of this type of amphibole in magmas and slags is dependent upon the presence of water or its vapour. The occurrence of potassium as in astrophyllite has already been explained as a normal impurity, in even the most pure sodium silicate. Considering the preponderance of titania, it is most astonishing that rutile did not appear as this is considered the most stable form of such titanium compounds, it must be assumed, therefore, that titania formed the main constituent of the glass, probably at first it was the only constituent, but as the normal melting point of titania is ca. 1,750° C., and glass first appeared with the disappearance of titania at ca. 1,200 C., it must be that the remaining constituents of the melt exerted in some way a catalytic action, possibly by eutectic formation. This explanation would be congruent with the employment of rutile in electrode coatings for the production of fluid slags of good covering power, whilst the rapid glass formation would, to a certain extent, reduce sputtering—these premises have been upheld by practical experiences. Further, the decrease in melting and reaction temperatures would produce a smooth-surfaced slag, and a consequent smooth surface to the weld metal—these are almost characteristic of electrodes containing ilmenite, and, as in the case of the slag here produced, ilmenite was one of the most prominent constituents, the production of a very smooth weld was not surprising. The known marked effect of manganese on the surface tension of slags and on the interfacial tension of slag and weld metal would no doubt be well based if more results were available of

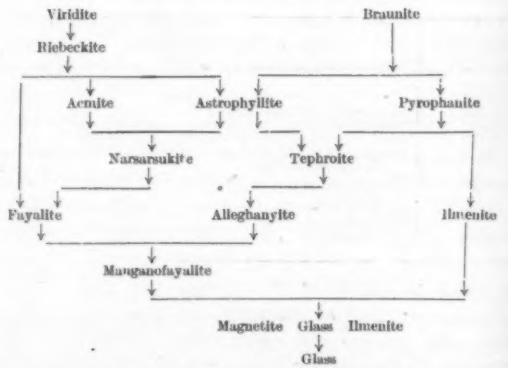

Fig. 7.—Probable sequence of mineral reaction and transformation.

the surface tension and other physical effects of manganese minerals in the molten condition. The utilisation of coatings containing high percentages of titania and manganese therefore would, by their inherent characteristics, correct any irregularities in welding caused by an unconformity in the iron-silicon ratio, thus we would anticipate that an electrode coating consisting almost entirely of pyrophanite with a small quantity of fayalite would exhibit the desired characteristics in an electrode of this type.

Conclusions

Although much work has been carried out on minerals' preparation and reaction at high temperatures, little work has been conducted on the physical properties of minerals at high temperatures. With the knowledge gained by a series of investigations on the mineral constituents of electrode coatings, and allied to information on the physical properties of the minerals produced by interaction at high temperatures, a surer foundation could be reached and a more scientific approach laid for the manufacture of welding electrodes based on fundamental knowledge.

11 Hatch and Wells. "Petrology of Igneous Rocks." London. 1926.

The Electron Microscope and its Application to the Study of Metals

By Robert G. Picard

R.C.A. Victor Division, Radio Corporation of America, Camden, N.J.

The past decade has seen the development of the electron microscope from a scientific curiosity into a tool of great practical worth. A huge field of direct visibility has been opened up, enabling us to see electron images of particles down to less than a millionth of an inch in size. More than a hundred times smaller than previously visible in the optical microscope. Here the principle of the electron microscope is briefly discussed and reference made to its applications in the study of metals.

WITHIN the past few years a new tool has been developed which extends the range of man's vision a hundredfold. This instrument, the electron microscope, has opened to direct observation a new realm which, a decade ago, was called sub-microscopic. Its applications have extended into many fields, and its contributions to the body of scientific knowledge have been substantial, especially in view of the fact that the history of the science of electron microscopy covers a period of only five or six years.

The reason for the rapid acceptance and wide application of the electron microscope lies in the fact that it is a new and more powerful eye for examining the sub-microscopic world. It does not in any way supersede the light microscope; it supplements the information obtainable by studying a realm of a different order of magnitude from that discernible by ordinary microscopy. Hence, the electron microscope becomes an important complement to the light microscope, the X-ray diffraction camera, the spectroscope, and all the other scientific instruments used in investigating the ultimate nature of matter.

The high useful magnifications attained with the electron microscope are a consequence of the great resolving power obtainable when a high velocity electron beam is used to irradiate a specimen. The resolving power of an optical instrument is the ability of the instrument to show detail. For a microscope, resolving power may be roughly defined as the smallest distance by which two points in an object may be separated and yet appear as two points in the image. Expressed mathematically, the definition of this smallest distance (the resolving power) is—

$$d = \frac{\cdot 61\lambda}{\text{N.A.}}$$

where λ is the wavelength of the illumination and N.A. is the numerical aperture of the lens system. The equation shows that the resolving power is proportional to the wavelength of the radiation used to illuminate the object. Visible light consists of radiations having wavelengths from .0004-.0008 mm. (4,000-8,000 Å), the shorter wavelength corresponding to violet light and the longer to red light. Details much finer than the wavelength of the illumination cannot be observed as the tool (light) is too coarse to make the desired measurements. Therefore, the resolving power of the light microscope is limited not by the mechanical perfection of the instrument, but by the fact that light is used to examine the specimen.

The useful magnification of the light microscope is limited to about 1,000 diameters if visible light is used for illumination and about 2,000 diameters if ultra-violet is used. This does not mean that photomicrographs cannot be enlarged to give over-all magnifications of a million times if desired, but such pictures will reveal no more than a 2,000-magnification picture would. Points just resolved at 2,000 diameters would be further separated at higher magnifications, but no additional detail would be revealed. This fact forces the microscopist to the conclusion that magnification is not the criterion which determines how deeply he can probe into the sub-microscopic world.

The electron microscope is not subject to the limitation imposed by the wave-length of light. With this instrument, specimens are irradiated with a beam of charged particles (electrons) travelling at very high velocity, and the theoretical resolution is unlimited down to sub-atomic dimensions. However, the aberrations inherent in present-day electron optical systems make it necessary to use numerical apertures about 1,000 times smaller than are used in light microscopes. In spite of this limitation, a hundredfold gain in resolving power over the light microscope has been accomplished, making possible useful magnifications of 100,000 diameters.

The beam of charged particles is focussed by electric or magnetic fields in a manner analogous to the focussing action of a glass lens on light. By reason of the analogy, the charged plates or magnets giving rise to the focussing fields are called electric and magnetic lenses. The electrons which irradiate the specimen and form the magnified image are scattered and stopped by the air in a fraction of an inch. So, in order to permit the electrons to reach the specimen and then form an image, the entire instrument must be highly evacuated. In fact, the electron microscope is simply a vacuum tube with means supplied to generate, accelerate, and focus electrons.

The electron beam used to examine specimens has extraordinarily low penetrating power. The common glass slide used for mounting specimens in ordinary microscopy is completely opaque to the beam, so that a different technique of specimen preparation must be used in electron microscopy. The usual technique, which has proved highly satisfactory during the past five years of pioneering in electron microscopy, consists of mounting the specimen on a thin nitrocellulose film supported by a 200-mesh wire screen. Such films may be made as thin as 100-300 Å, are transparent to the

electron beam and show no structure of their own. Particles suspended on the film over the holes in the wire mesh may thus be studied against a clear background.

Not only must the specimen support be extremely thin, but the specimen itself should be less than one micron in thickness if internal details are to be demonstrated with the instrument. Bacteria and colloidal particles are ideal electron microscope specimens; others require the application of a great deal of ingenuity and labour before they may be studied satisfactorily.

The most widely used electron microscopes have magnetic lenses and an optical system analogous to that of the ordinary compound microscope (Fig. 1). These features have been embodied in extremely versatile research instruments such as that illustrated in Fig. 2, and more compact, desk type models (Fig. 3). Both instruments are capable of resolving powers better than 100 Å, and include high-speed vacuum pumping systems and reliable power supplies which permit rapid operation and taking many electron micrographs per day.

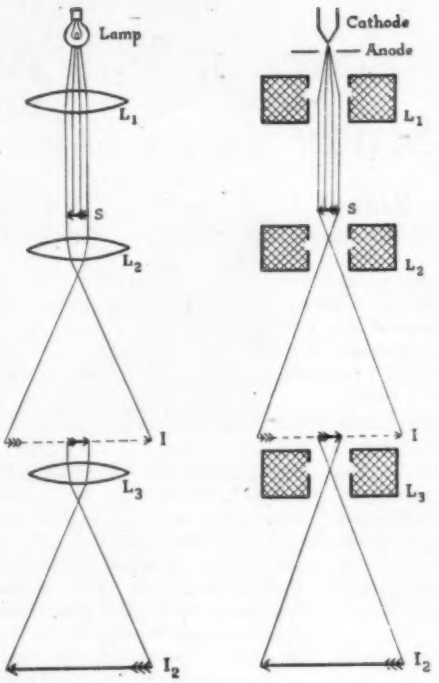

Fig. 1.— Optical analogy between light and electron microscopes. L_1 substage condenser, L_2 objective lens, L_3 eye piece or projection lens, S specimen.

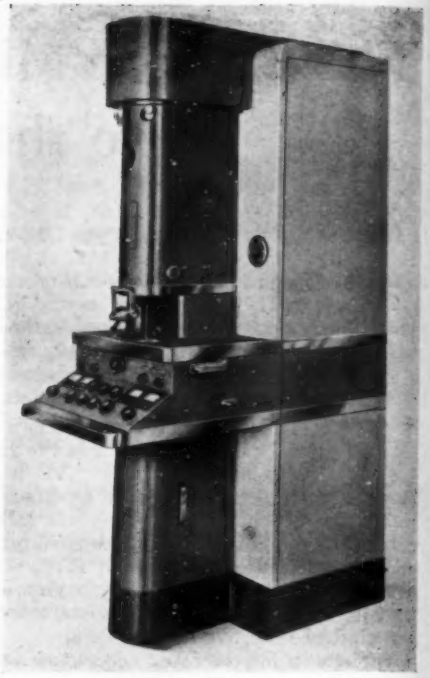

Fig. 2.—Universal Electron Microscope.

The applications of the electron microscope to the study of metals dates back nearly a score of years to the earliest development in electron microscopy. One of the first subjects ever studied by electron optical means was the surface of electron emitting materials, especially incandescent metal surfaces. The instrument, called an emission microscope, formed an image of the electron emitting cathode at magnifications up to several hundred diameters. From these pictures information was obtained about the effects of activators added to electron emitters, the effects of cold working, and related data.

Modern, high magnification electron microscopes have contributed much information to many phases of metal technology. Colloidal particles, distinguishable only as dots with the light microscope, may now have their sizes and shapes determined and crystal structures verified. Metallic smokes may be collected readily on specimen screens and examined (Fig. 4). Evaporated metals, when deposited on thin films, are ideal specimens for electron microscopy, and the structures of films only a few atoms thick have been determined. (Fig. 5).

One of the most important phases of the study of metals is that involving the study of the structure of metallic surfaces, that is, metallography. Unfortunately, the specimens used are of such thickness as to be opaque to the electron beam in an electron microscope, and no method is known for making metallic specimens thin enough for use in the instrument. However, the possibilities of the electron microscope were so appealing to metallographers that they inspired the development of a number of very successful

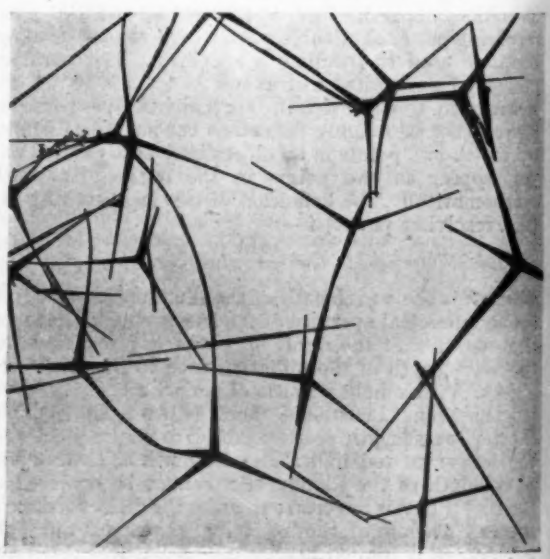

Fig. 4.—Zinc oxide smoke.

Fig. 3.—Console Electron Microscope.

techniques for making accurate replicas of metal surfaces that were sufficiently thin for use in high power instruments.

The simplest replica method consists of coating the prepared surface with a thin film of collodion or lacquer, and then stripping this film from the surface by mechanical means. Such a replica is a negative of the surface and, with careful preparation, will give a faithful reproduction. However, these replicas suffer from a lack of contrast which is manifested by a lack of sharpness and detail in the microscope image.

Recently, the usefulness of such replicas has been greatly enhanced, and the information obtainable increased by depositing a thin film of heavy metal on the replica. If the metal is evaporated in *vacuo* and strikes the replica at a low angle, elevations in the replica will cast shadows. Such a metallic film, if only a few Ångstrom units thick, will not be opaque to the electron beam, but its high scattering power will make the uncoated shadows more evident and thus increase contrast. (Fig. 6).

Several methods have been evolved for making positive replicas. One of the earliest of these consisted of first evaporating a thick film of silver on to the surface, stripping this mechanically, then flowing collodion over this negative and, finally, dissolving the silver with acid leaving the collodion positive reproduction of the surface.

Another method, widely used for the study of aluminium and other metals which oxidise readily, consists of oxidising the prepared metal surface to a small depth, and then chemically separating the oxide coating from the underlying metal. This oxide film can be made sufficiently thin to be examined in the electron microscope and is not merely a replica of the surface, but is the actual surface. (Fig. 7).

Probably the most widely used method of making replicas is a two-step, positive replica technique using polystyrene and silica. Polystyrene is moulded against the metal surface at high temperature and pressure. When cool, the two are separated, mechanically if possible, or by dissolving the metal if necessary. The polystyrene negative is then placed in a vacuum chamber and a very thin film of silica is evaporated on to it. Silica has a high mobility over polystyrene, and covers

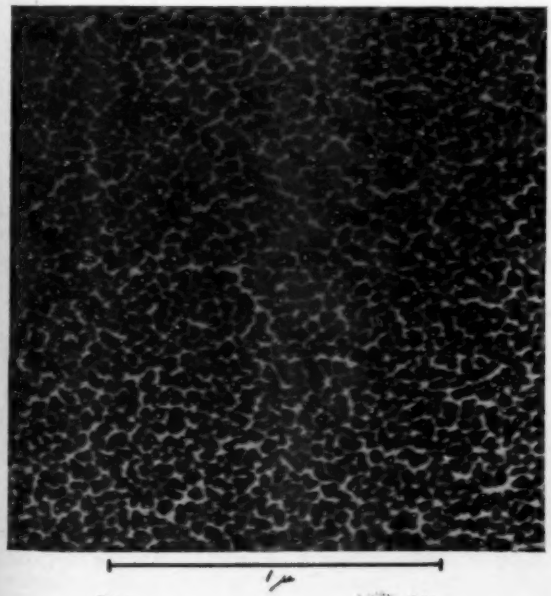

Fig. 5.—Evaporated gold.

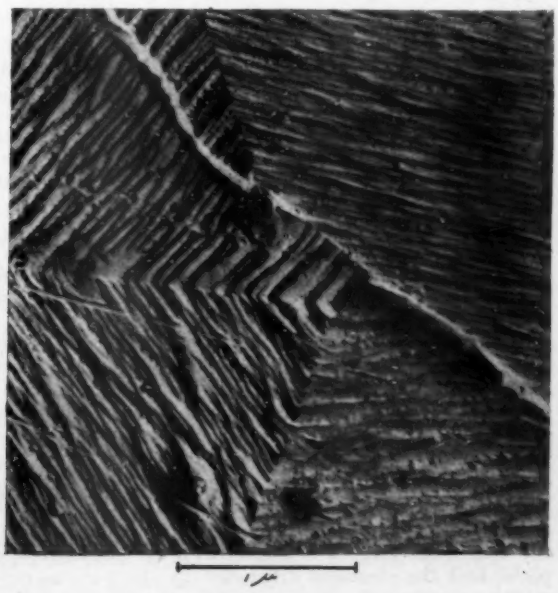

Fig. 6.—Chrome-iron alloy. Collodion replica shadowed with a thin film of chromium.

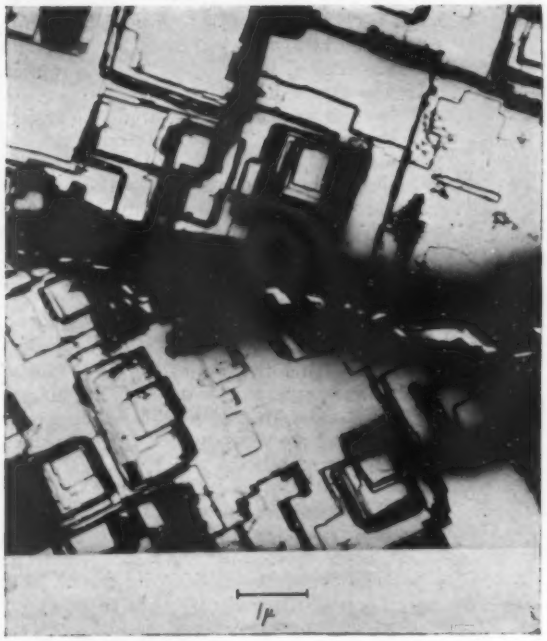

Fig. 7.— Oxide film showing cubic block structure adjacent to a grain boundary in a deep-etched sample of an annealed aluminium alloy sheet.

the surface uniformly. The polystyrene is finally dissolved, leaving a thin silica positive replica of the surface which shows no structure of its own, but bears an accurate reproduction of the original metal surface. (Fig. 8). All of the positive replicas may also have their contrast enhanced by metallic shadow casting.

Because of its small numerical aperture, the electron microscope has a depth of focus that is great compared to that of the light microscope. This feature makes possible the making of stereoscopic micrographs by photographing the specimen field, tilting the specimen, and photographing the same field from the new aspect. The heights of surface projections and depth of etch can thus be measured at magnifications of many thousands of diameters and with a resolution of the order of 150 Ångstrom units. (Fig. 9).

Size, shape, and density variations of specimens are easily determined with the electron microscope, but the chemist, physicist, and metallurgist are also interested in the atomic structure of materials. The electron microscope has the well regulated power supply, the lens system, and the photographic facilities required for an electron diffraction camera which will permit studies of atomic structure. For this reason, the universal instrument has facilities for producing electron diffraction patterns. Incredibly small amounts of materials may be used and clear, sharp patterns obtained. To use the electron microscope as a diffraction camera, the electron beam is allowed to strike the specimen, but is not focussed to produce an image of the specimen. Instead, the source of electrons is focussed on the photographic plate, and the electrons diffracted by the various crystalline planes of the specimen strike the plate at different points. Each material has a characteristic pattern which serves to identify it.

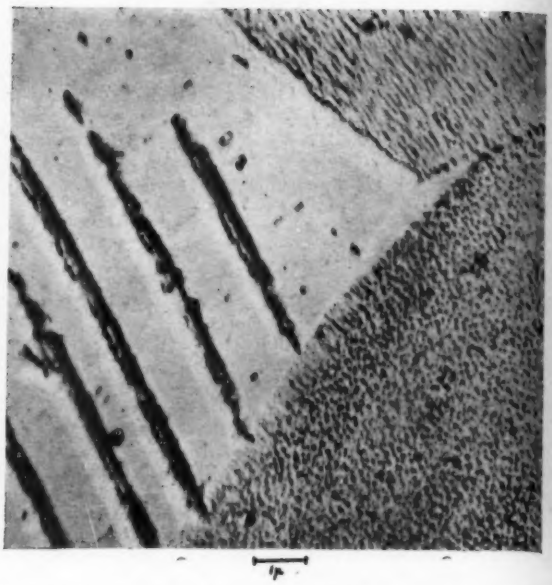

Fig. 8.—Silica replica of stainless steel.

Diffraction patterns may be obtained by passing the electron beam through a thin film of the specimen (Fig. 10a) or by allowing the beam to strike the surface at grazing incidence (Fig. 10b). This latter procedure gives rise to reflection patterns, which are actually produced by the passing of the beam through minute projections above the surface of the specimen. This is an extraordinarily powerful technique for studying the structure of surfaces, because the low penetrating power of the electron beam assures that the pattern will be due to the material on the surface and not to the backing substance.

Many hundreds of beautiful electron micrographs of metals have been made, and yet the application of the electron microscope to the study of metals is still in its infancy. A number of fundamental problems await solution and clarification. Replicas are easily made with a little practice, but the interpretation is based on the assumption that one side of the replica is flat and smooth. However, it is quite generally agreed that this condition does not exist in many cases. Attempts must be made

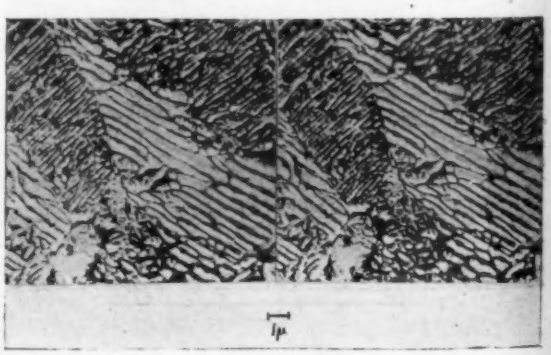

Fig. 9.—Silica replica of pearlite. Stereoscopic pair.

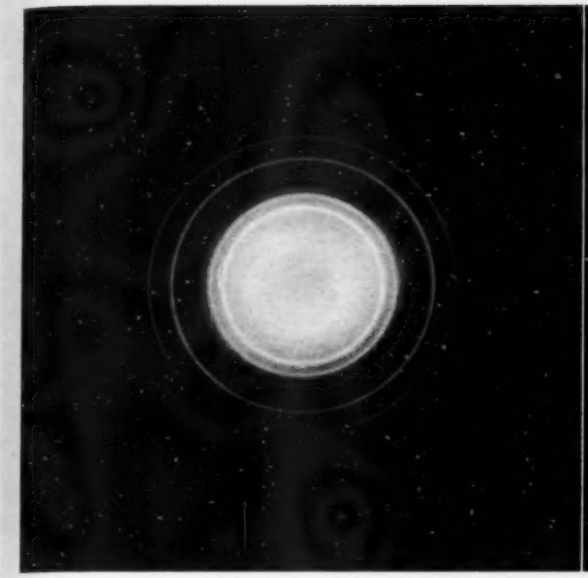

Fig. 10a.—Transmission diffraction pattern of aluminium.

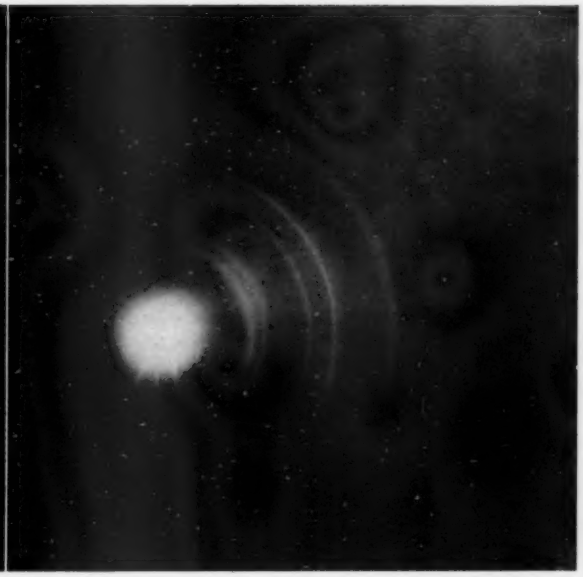

Fig. 10b.—Reflection diffraction pattern of silver surface.

to find materials and develop methods which will more nearly satisfy this assumption.

Furthermore, allowance must be made for distortion in replicas. A great deal of checking of this point has been done using diffraction gratings as samples, and proponents of the polystyrene-silica technique feel that they have demonstrated that this procedure produces less distortion than others. Nonetheless, there is considerable debate on the subject. The answer to such questions may well determine whether simple stripping methods can compete with the more complicated two-step processes.

A few years ago, a scanning electron microscope was invented, but its development was halted temporarily by the war. This instrument systematically scans a metal surface with an electron beam and produces a good picture of the surface in a manner analogous to the production of a television image. At the time when work was interrupted, the resolving power of the scanning microscope did not equal that obtainable with replicas in transmission type instruments, but its further development may do much to eliminate replica methods. Of course, those who use their microscopes for many types of work besides metallography will still need to use replicas in this field.

Many techniques, developed exclusively for the electron microscopy of metals, have been carried over to other branches of the science. Replicas have been used to study dental specimens and some organic materials. Thin silica films are often used to replace nitrocellulose in chemical and bacteriological work, as they do not break as easily and will withstand high temperatures. The shadow casting method has recently been found very useful in the study of bacteria and viruses. This technique made visible a confusing structure in the collodion supporting film, so a method was developed to make replicas of viruses and bacteria using polished glass as a background which exhibits no structure. Thus, in ways such as these, the limits of observation are being pushed back, and this new tool, the electron microscope, is becoming more useful as the skill and ingenuity of its users grow.

Forming of Aluminium Alloys by the Rubber Die Press

THIS 32-page Bulletin, with numerous illustrations and diagrams, opens with an introduction to the principles of the process, and a description of the latest types of rubber die presses and how they are used.

The process will aid manufacturers who need to produce quickly components varying in size and shape, but are not required in quantities that warrant the use of hardened steel tools and dies. The rubber die process is normally carried out with simple and relatively inexpensive tools, which can be produced without elaborate tool-room plant and craftsmen.

The versatility of the process is shown by an illustration of the largest single component being produced upon the smallest press—by using the full capacity of the platen for a single pressing.

A considerable portion of the booklet is devoted to the technique of rubber die press work. The methods detailed are: Blanking and piercing (together with the essential press requirements); flanging in its various forms, with graphs showing the respective concave and convex flanges that are permissible without wrinkling or splitting; how moderate drawing operations can be achieved with the aid of supplementary rubber pads; and means of overcoming springback in heat-treated alloys, together with useful tables of springback angles for simple straight flanges.

Detailed instructions are given on die design, and how to make temporary or permanent dies and the ancillary tools in various materials, such as hardwood, zinc alloys, mild steel, cast iron, etc.

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Grain Boundary Penetrations by Liquid Metals

I.—Some Service Failures

by W. R. Smith, B.Sc., and P. E. J. Forsyth

Metallurgists, Royal Aircraft Establishment, Farnborough.

Grain boundary penetration by a molten metal is not uncommon in present-day engineering practice and, since such penetration does destroy the intercrystalline cohesion of an alloy, premature failure can and does occur from this cause. The susceptibility to penetration of an alloy depends upon the nature of the alloy and of the liquid metal in contact with it. In this article some examples of penetration in copper, aluminium, and ferrous alloys are discussed.

ALTHOUGH the illustrations of failures discussed in this article were recorded over a period of some years, the penetration of liquid metals is by no means uncommon in present-day engineering practice, and many of the usual alloys, both ferrous and non-ferrous, are susceptible to this type of failure. Under normal conditions, the crystal boundaries of these alloys do not suffer selective attack, but when they are subjected to tensile stresses in the presence of liquid metal, they are preferentially attacked. The susceptibility to penetration of an alloy is dependent on the nature of that alloy and also of the liquid metal, and this article deals with some examples of penetration in copper, aluminium, and ferrous alloys.

The true cause of the penetration of the grain boundaries is not fully understood, but it is known that the phenomenon is closely linked with stress corrosion cracking. This is brought out very clearly in that one of the tests for revealing susceptibility to season cracking is to immerse the material in a solution of mercurous nitrate. If the material is susceptible to this type of cracking, it will develop intercrystalline cracks caused by penetration of mercury along the grain boundaries. The nature of a crystal boundary is known to differ from that of the crystal, and is believed to have an atomic arrangement transitional between the lattice structures of the abutting crystals, i.e., the grain boundary is a transitional layer resulting from a compromise between the atomic forces of the neighbouring grains, and so the boundary atoms are not so closely bound as the atoms in the interior of the grains. The disturbed nature of the crystal boundaries is shown by the fact that diffusion of one metal in another is much faster through the grain boundaries than through the normal crystal lattice, but it must be pointed out here that those liquid metals which cause failure by penetration of the grain boundaries in most cases have negligible solubility in the normal lattice of the attacked materials.

The essential factors which contribute to failure by grain boundary penetration are :

(1) The nature of the material and of the molten liquid.
(2) The presence of tensile stresses. These can either be applied externally or be present as internal stresses across the grain boundary.

The remainder of this article deals with some examples of service failures together with a general discussion of the factors mentioned above. For simplicity, the alloys are divided into the three main groups of engineering alloys.

Copper Alloys

The intercrystalline failure of copper alloys is a very important subject, and the two most usual causes are season-cracking and the intercrystalline penetration by molten metals. These two phenomena are closely related, and all the evidence indicates that the high-tensile brasses are the most susceptible materials to penetration by molten metal.

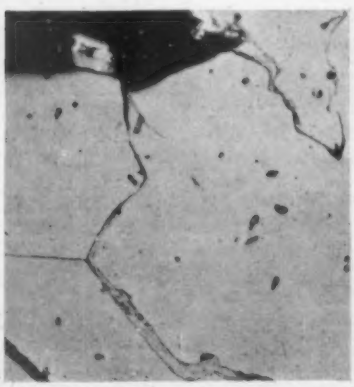


Fig. 1.—Lead-tin solder in high strength beta brass casting. (× 100).

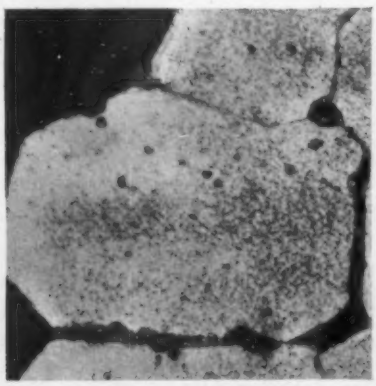


Fig. 2. (× 1,500). Lead in grain boundaries of beta brass union nut.

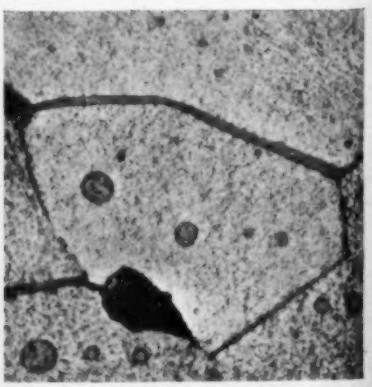


Fig. 3. (× 1,500).

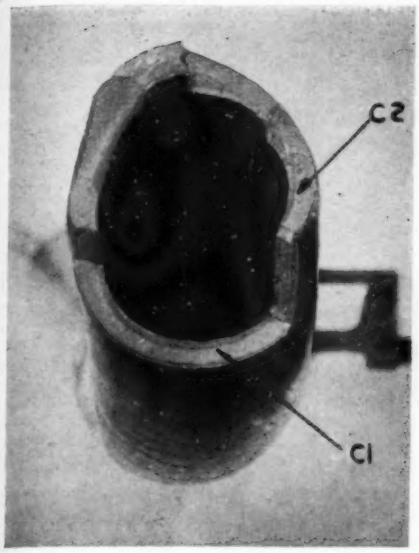


Fig. 4. Fracture of 100 tons/sq. in. steel tube (× half).

Soldering operations on copper alloys are carried out extensively in engineering practice, and so considerable discretion and care must be used when tinning these alloys. The microstructure of the alloy is an important consideration, and the high-copper content alloys have been shown to be the least susceptible to penetration by liquid metal. The alloys with a completely beta structure are the most susceptible to attack and the high-strength brasses with this type of structure are very prone to failure. The susceptibility to attack is greatly increased by the addition of aluminium to the brass. A particular illustration of this effect was given by the failure of a high-strength brass casting of the composition—

Cu	*Zn*	*Mn*	*Al*	*Sn*
60·0%	35·4%	1·46%	2·78%	0·18%

The percentages of the addition elements, particularly the aluminium, were such as to give the material a "fictitious-zinc" content of about 47%, so taking the microstructure into the completely beta range.

The casting was found to be cracked seriously after tinning, and on microscopic examination it was found that the beta grains were enveloped by solder which had penetrated during the tinning operation. In this case the stresses were internal and were so-called thermal stresses.

Fig. 1 is a photomicrograph showing the solder penetration in the casting.

A similar high-strength casting of the composition—

Cu	*Zn*	*Mn*	*Al*	*Sn*
60·5%	35·6%	1·66%	1·62%	0·39%

did not suffer penetration, and the microstructure was duplex in character.

An interesting variation of grain boundary penetration was seen in the case of a leaded brass where the lead had penetrated the grain boundaries. Here again the microstructure was completely beta.

In this particular instance, union nuts in leaded brass to specification B.S. 249 had cracked when tightened down, and on microscopic examination it was found that the lead had formed thin films at the grain boundaries. The lead which should have existed as discrete globules had migrated to and penetrated along the grain boundaries when the material had been annealed.

Fig. 2 is a section through the fracture, and Fig. 3 shows more clearly the lead at the grain boundaries of the beta solid solution.

Nuts manufactured in similar material but showing an $\alpha + \beta$ microstructure did not show this effect.

Ferrous Alloys

Some steels are also susceptible to penetration by liquid metals, and here again, as in the copper alloys, those alloys most susceptible are the high-strength alloys. The most usual metals which penetrate the grain boundaries of steels are solders and brazing alloys.

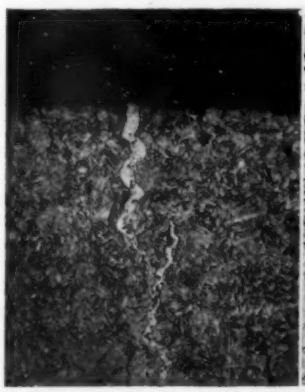


Fig. 5.—Solder penetration of 100 tons/sq. in. steel (× 100).

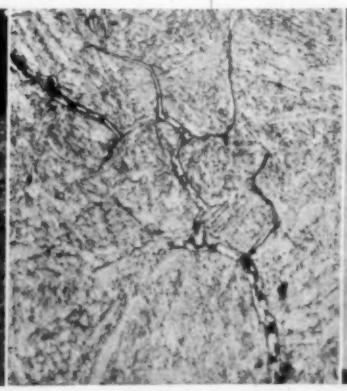


Fig. 6.—Solder at grain boundaries of 100 tons/sq. in. steel tube (× 500).

Fig. 7.—Penetration of stainless steel sheet by copper-zinc brazing alloy (× 30).

A recognised method of bending tube without its collapsing is to fill the inside with "solder" before bending. This acts as a former and can be run out after the bending operation. This practice was found in some instances to contribute to the failure of high-tensile nickel-chromium steel tubes, failure occurring in service when these tubes were subjected to stress.

The cause was found to be weakness of the tubes produced by intercrystalline penetration of the "solder." The exact stage at which penetration took place could not definitely be stated, but it was probably initiated after the bending when the "solder" was melted out, and propagated during the subsequent heat-treatment of the tubes.

In Fig. 4, the extent of the cracks caused by penetration are indicated by the arrows C1 and C2, and Figs. 5 and 6 show the nature of the solder penetration through the grain boundaries.

The steel had been heat-treated to give a tensile strength of 100 T/in.² and had the composition—

C	*Si*	*Mn*	*Ni*	*Cr*
0·31%	0·27%	0·47%	4·23%	1·21%

The stresses necessary for penetration in this case were caused by the retention of the martensitic structure by quenching.

There have been many instances in the past of molten brasses and copper-zinc brazing alloys causing failure by their intercrystalline penetration, and in many cases the cause has been attributed to the high internal stress set up by steep temperature gradients produced by rapid heating.

Fig. 7 shows the intercrystalline penetration of austenitic steel sheet by a copper-zinc brazing alloy. The opening out of the main crack by the stress is shown very clearly, and in fact the sheet took up the form of an arc.

Aluminium Alloys

Some modern high-strength aluminium alloys are supposed to be very susceptible to stress corrosion, but very little data has been published on their susceptibility to penetration by liquid metals, and the authors were very interested to find intercrystalline penetration of an Al-Cu-Mg-Zn alloy by molten Wood's metal. A section of extrusion in the Al-Cu-Mg-Zn alloy was mounted in Wood's metal in order to do hardness determinations, and on remounting for microscopical examination it was found that the Wood's metal had penetrated the section where the residual stresses were expected to be the greatest. This effect is shown in Fig. 8 and the intercrystalline nature of the penetration is shown in Fig. 9.

A type of defect sometimes found in aluminium alloys is overheating, and although this does not strictly come within the scope of this article, it justifies its inclusion in that it is analagous to the failure of the brass union nut reported above.

Here the intercrystalline cohesion of the material is broken down by films of low melting point constituents, e.g., $CuAl_2$, in the grain boundaries. Fig. 10 is a photomicrograph of a section through the fracture of an overheated duralumin type of alloy, and shows some of the $CuAl_2$ network which embrittled the material.

These examples show the dangers of allowing contact between molten metal and stressed parts in service, but these conditions are very likely to occur during soldering and brazing operations. Where it is impossible to prevent contact of a part with liquid metal, the component should be thoroughly stress-relieved. This can easily be achieved in the case of brasses by heating to about 300° C. Where there must from necessity be contact between an externally stressed part and molten metal, then the only preventative against failure by intercrystalline penetration (if the material is susceptible to this type of attack) is to replace the liquid metal by one which does not so easily attack the grain boundaries, e.g., silver-bearing solder such as lead-silver.

The correlation that may be drawn between the order of penetration of liquid metals and their ability to penetrate capillary cracks seems to offer some light on the mechanism of penetration, and it may be that some balance between chemical reactivity and penetrating ability may be the governing factor.

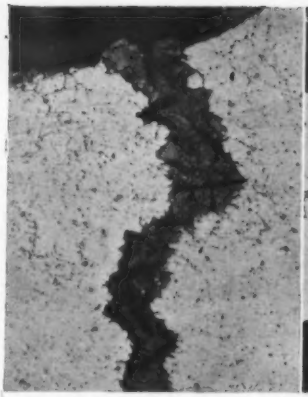


Fig. 8. (×40). Wood's metal penetration of high-strength aluminium alloy.

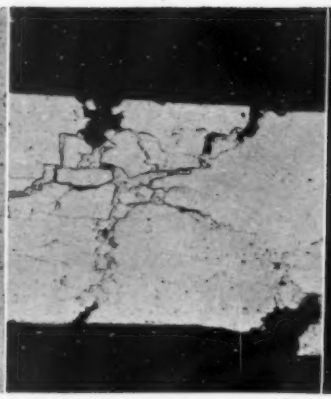


Fig. 9. (× 30.)

Fig. 10.—Section through fracture of overheated duralumin (× 500).

Hands Full of Power

THE last few decades have seen a tremendous development in the use of power, not only in its application to the big tasks of engineering, but in fields associated with operations that formerly involved much manual labour. In almost every walk of life power is now being successfully applied to small operations and work of a laborious nature is gradually being iradicated. The development and application of electricity is mainly responsible for this increasing use of power.

In no field has the benefits of electric power been more evident than in its application to tools. This is shown in a vivid manner by a full colour sound film recently shown in London in which S. Wolf & Co. Ltd. describe how their electric tools are made, and show their many uses. The film, which has been made by United Motion Pictures, Ltd., with commentary spoken by John Snagge and David Lloyd James, occupies twenty-two minutes in showing, and it is both interesting and informative.

The film opens with scenes depicting the harnessing and control of electricity and is followed by a sequence of some of the tools which provide industry with a means for substantially reducing old laborious production methods—drills, sanders, hammers, grinding and polishing machines, screwdrivers, etc., all tools for the unskilled as well as the skilled, and for doing work with great accuracy, more economically, and in a fraction of the time.

S. Wolf & Co. Ltd. are to be congratulated on their enterprise, and those responsible for the photography and the commentary are to be commended for the preparation of a film which has considerable educational value.

Die Steels for Hot Work

By D. W. Rudorff, M.Inst.F.

The need for conserving alloying elements in short supply during the war led to many investigations as a result of which modifications were made in the compositions of alloy steels for particular services. In several countries the possibilities were explored of limiting the percentage of tungsten in steels used for hot work dies and punches, and, although information regarding the work carried out has been fragmentary, two recent investigations are of considerable importance, a review of which is given in this article.

DURING the war the demand for alloying elements far exceeded their availability, and numerous investigations were carried out in an effort to reduce, or to practically eliminate the use of those elements in short supply. From the results of these investigations many alloy steels were produced as substitutes for those commonly used when it was possible to obtain the elements used in their composition. In some instances these substitute alloy steels have been proved to be comparable in many respects with those they have displaced, and it is probable that they will be standardised for specific types of service. For certain purposes the provision of substitute alloy steel has not been quite so successful. In Germany, for instance, the desire to conserve tungsten led to investigations designed to eliminate or considerably reduce the percentage of tungsten in steels for hot work dies and punches, etc.

Progress in Die Steels for Hot Work

Numerous investigations have been made in the past into the possibility of either completely eliminating or considerably reducing the percentage of tungsten in steels used for hot work dies and punches, etc. While it was recognised that war-time needs gave a particular impetus to the development of substitute steels in Russia and Germany, information on the work carried out in these countries has been fragmentary and sketchy. A brief resumé of outstanding work in these two countries may therefore prove valuable.

By far the most ambitious attempt at a systematic study of the influence of the tungsten percentage in tungsten-chromium die steel for hot work and the complete or partial replacement of the tungsten content by molybdenum and/or vanadium was the investigation carried out in Germany by Hopage, Völka, and Tinti.* Their work was mainly concerned with properties displayed by the steel tested at 500–600° C., and with the ability of the steel to withstand the abrupt changes in temperature to which hot work dies are exposed in the production process. Quite generally, a steel to be suitable for hot work must possess high resistance to softening, and must exhibit high strength at the working temperature concerned. Another factor to be considered in the development of a steel of this kind is good thermal conductivity of the steel which is important with regard to the prevention of destruction of the tool by fire checking.

The steels investigated by the aforementioned workers were of compositions ranging from 0·22–0·42% C, 1·3–2·1% Cr, 0–4·9% Mo, 0–1·2% V, and 0–11·7% W. The actual compositions are listed in the subjoined Table I, where the steels are seen to be subdivided into 10 main groups. The different heats (of 10 kg. each) were prepared in an electric resistance furnace. After forging into rounds of 25 mm. dia. the pieces were heated at 700° C. for two hours. From each round seven discs of 10 mm. thickness each were cut and quenched in oil from 900°, 950°, 1,000°, 1,050° 1,100°, 1,150°, and 1,200° C., respectively. These were then fractured. Upon inspection of the fractures, the fractured pieces were tempered for 30 mins. at 400°, 450°, 500°, 550°, 600°, and 650° C., respectively. For the steels comprising groups No. 1 (tungsten steel) and No. 2 (molybdenum steel) the optimum quenching temperature was found as 1,000° C. Of group No. 1 the 6% tungsten steel exhibited greatest hardness after quenching from 1,000° and 1,050° C., while the steels with higher tungsten percentage in that group required quenching temperatures from 1,150°–1,200° C. to develop their maximum hardness. Almost identical hardness values were obtained by quenching the vanadium steels comprising group No. 3 from 1,150°–1,200° C.; their hardness was found to decrease progressively with lower quenching temperatures. There exist distinctly different hardening characteristics in multi-alloy steels for the hardening ranges of 1,050°–1,200° C. temperature of quench, and of 900–1,000° C. temperature of quench, respectively. A quenching temperature of about 1,050° C. was found to yield optimum hardness values.

These findings were confirmed by fracture tests and by micrographic examination. The fracture of steels comprising group No. 1 when quenched from 1,200° C. revealed coarse-grained structure throughout, typical of overheating. A tungsten content of 5% did not prove sufficiently high to entirely eliminate the danger of overheating. Similarly, a molybdenum content of 3% (group No. 2) did not give satisfaction in this respect; and the same applies to the 5% molybdenum steel of group No. 10. The steels No. 52 (group No. 7c.) and No. 64 (group No. 8c) with 3% Mo–1% V and 6% W–1%V, respectively, exhibited cleavage planes through the range of quenching temperatures from 900°–1,200° C.

With regard to the influence of the tempering temperatures upon the hardness of specimens quenched from 1,150° C. in oil, a noticeable increase in hardness takes place at tempering temperatures ranging from 550°–600° C. In the comparative investigations, as far as they were concerned with strength at elevated temperatures with the sensitivity to fire checking, and

* *Arch. Eisen, H. W.*, vol. 17, 1943, p. 57–64

TABLE I.

Group	Steel No.	C.	Cr. Per Cent.	Mo. Per Cent.	V	W
Basic Material	1	0·26	1·55	—	—	—
1	2	0·23	1·55	—	—	1·06
	3	0·24	1·31	—	—	1·98
	4	0·24	1·31	—	—	3·05
	5	0·24	1·31	—	—	6·02
	6	0·30	1·55	—	—	8·0
	7	0·32	1·61	—	—	11·74
2	8	0·26	1·55	0·51	—	—
	9	0·26	1·45	0·90	—	—
	10	0·23	1·45	1·40	—	—
	11	0·30	1·61	2·05	—	—
	12	0·30	1·60	2·90	—	—
3	13	0·29	1·55	—	0·20	—
	14	0·22	1·38	—	0·36	—
	15	0·24	1·55	—	0·64	—
	16	0·28	1·55	—	1·16	—
4a.	17	0·20	2·05	0·44	—	3·00
	18	0·27	1·85	0·92	—	3·17
	19	0·28	1·57	1·90	—	3·05
	20	0·30	1·52	2·95	—	3·32
4b.	21	0·29	1·57	0·50	—	5·00
	22	0·32	1·55	0·86	—	4·88
	23	0·29	1·47	1·95	—	4·72
	24	0·26	1·42	2·95	—	5·00
5a.	25	0·30	1·45	—	0·07	3·08
	26	0·27	1·47	—	0·41	3·10
	27	0·29	1·45	—	0·78	3·10
	28	0·25	1·42	—	1·20	2·70
5b.	29	0·26	1·48	—	0·22	5·13
	30	0·27	1·48	—	0·36	5·07
	31	0·27	1·50	—	0·70	5·06
	32	0·26	1·53	—	1·10	4·80
6a.	33	0·31	1·35	0·50	0·20	—
	34	0·26	1·59	0·52	0·37	—
	35	0·28	1·59	0·60	0·65	—
	36	0·26	1·38	0·47	1·05	—
6b.	37	0·32	1·59	0·95	0·26	—
	38	0·26	1·45	0·98	0·35	—
	39	0·28	1·70	0·93	0·60	—
	40	0·30	1·59	0·95	1·22	—
7a.	41	0·26	1·83	0·85	0·11	2·60
	42	0·30	1·50	0·88	0·42	2·76
	43	0·26	1·59	0·86	0·66	3·03
	44	0·29	1·40	1·00	0·95	3·02
7b.	45	0·24	1·27	1·95	0·16	3 11
	46	0·27	1·77	1·95	0·41	3·10
	47	0·30	1·70	1·95	0·60	3 25
	48	0·31	1·34	1·90	1·00	2·97
7c.	49	0·26	1·37	3·30	0·17	2·94
	50	0·28	1·43	2·80	0·40	3·33
	51	0·26	1·58	2·60	0·61	3·03
	52	0·23	1·27	2·90	0·99	3·06
8a.	53	0·25	1·32	1·15	0·18	4·70
	54	0·22	1·83	0·95	0·60	4·64
	55	0·26	1·43	0·95	0·63	5·00
	56	0·27	1·40	1·10	1·05	5·14
8b.	57	0·25	1·35	1·90	0·24	4·70
	58	0·22	1·50	2·00	0·41	4·72
	59	0·27	1·47	1·95	0·55	4·75
	60	0·27	1·38	2·00	0·95	4·76
8c.	61	0·25	1·35	2·90	0·16	4·96
	62	0·27	1·38	2·90	0·3	4·77
	63	0·28	1·35	2·90	0·57	4·80
	64	0·26	1·31	2·85	0·98	6·02
9	65	0·28	1·43	—	—	4 68
	66	0·30	1·36	—	—	4·45
	67	0·36	1·53	—	—	5·43
	68	0·41	1·53	—	—	4·40
10	69	0·24	1·50	4·85	—	—
	70	0·29	1·50	4·70	—	—
	71	0·34	1·50	4·15	—	—
	72	0·42	1·42	4·90	—	—

with the thermal conductivity of the various steels, a uniform quenching temperature of 1,150° C. was adopted, and the respective elongation at fracture and notched bar impact strength data were based upon an U.T.S. of 88·9–95·3 tons per sq. in. (approximating to 44–45 Rockwell C hardness). From the data obtained on the steels containing either W, Mo, or V as sole alloying constituent in addition to Cr, the influence of each of the three aforementioned alloying constituents upon the physical characteristics of the steel can be charted separately, as shown in Figs. 1, 2, and 3. On the percentage basis vanadium is seen to have the greatest influence upon the mechanical strength, molybdenum ranking next, and tungsten taking the last place. Thus, for instance, a 0·2% proof stress of 50·8 tons per sq. in. at 400° C. can be obtained by adding to steel No. 1 either 0·5% V, or 0·9% Mo, or 4·4% W. It goes without saying that it is not permissible to establish any generally valid rules concerning the percentage ratio of this basis. However, these figures can serve as a rough indication of the relative influences of the aforementioned three alloying elements.

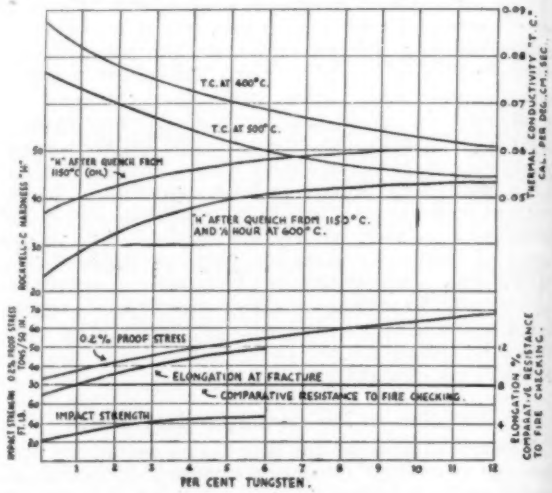

T.C. AT 400°C.
T.C. AT 500°C.
"H" AFTER QUENCH FROM 1150°C (OIL)
"H" AFTER QUENCH FROM 1150°C. AND ½ HOUR AT 600°C.
0.2% PROOF STRESS
ELONGATION AT FRACTURE
COMPARATIVE RESISTANCE TO FIRE CHECKING.
IMPACT STRENGTH
ROCKWELL-C HARDNESS "H"
IMPACT STRENGTH 0.2% PROOF STRESS TONS/SQ. IN. FT. LB.
THERMAL CONDUCTIVITY °F C. CAL. PER SEC. CM. SEC.
ELONGATION %. COMPARATIVE RESISTANCE TO FIRE CHECKING.
PER CENT TUNGSTEN.

Fig. 1.—Influence of tungsten upon the properties of 0·25/0·30 C—1·5 Cr Steel.

With regard to the effect of vanadium additions, it is necessary to note the considerable deterioration in the notched bar impact strength caused by increasing vanadium percentage in the alloy. But it is not at all unlikely that the situation will be quite different in the case of a higher carbon percentage.

The determination of the ability of the steel to resist fire checking requires the employment of a special laboratory device capable of reproducing conditions similar to those prevailing in practice. The task (of devising an apparatus of this kind, is, however, made difficult by the fact that fire checking is due to the joint effect of a number of inherent properties of the material which, in combination with influences originating from operating conditions, produce that phenomenon. Fundamentally, two groups of basic properties of the material can be considered to be responsible. The first of these comprises the properties which produce or aggravate the stresses constituting the cause of fire checking (outstanding among these are low thermal conductivity of the material and large thermal expansion); while the other group includes such properties as loss of strength with increasing temperature, embrittlement, and insufficient corrosion resistance. In view of the difficulties involved in reproducing actual service conditions, it must be considered sufficient for

comparative purposes to employ a device of the kind outlined in Fig. 4, where the specimens carried on a revolving drum are alternatively heated to dull heat by a gas flame, and subsequently cooled by a water jet.

In the investigation under review each specimen was in this way subjected to 1,000 heating and cooling cycles. Obviously, the tendency to fire checking is related to the shape and the size of the piece, as well as to its physical properties, and a number of different shapes were therefore investigated in order to find the one best suitable for this testing procedure. A specimen (1) of roll shape, as shown in Fig. 5, proved unsatisfactory, but shape (2) gave satisfactory results. Still better results were obtained with the small sphere (3). In view of the fact that the large roll (4) was found to require unduly long heating time, shape (3) was chosen as standard shape. These tests confirmed the well-known fact that tungsten steels possess by far the greatest resistance to fire checking. The beneficial influence of tungsten in this respect was noticeable in the case of the materials containing other alloying constituents in addition to tungsten.

In the tests referred to, the determination of the thermal conductivity was also carried out on a comparative basis. In this method rods of 6 mm. dia. and 120 mm. length were dipped at one end into a salt bath of given temperature, and the temperature was measured at the other end after the heat loss from the rod had attained equilibrium. The thermal conductivity was then established by evaluating the temperature indication as compared to that obtaining with a rod of known conductivity. This method was found to be sufficiently accurate for the purpose concerned. The variation in thermal conductivity with the percentage of alloying constituent of several of the steels investigated is charted in Figs. 1–3. It will be noted that tungsten exerts a smaller influence upon the thermal conductivity than either molybdenum or vanadium. Up to contents of 1% the respective effects of molybdenum and vanadium are seen to be practically identical. The average decrease in thermal conductivity if the temperature is raised from 400°–500° C. amounts to some 0·0004 cal. per °C. cm. sec., and the decrease is the smaller the lower is the thermal conductivity of the steel.

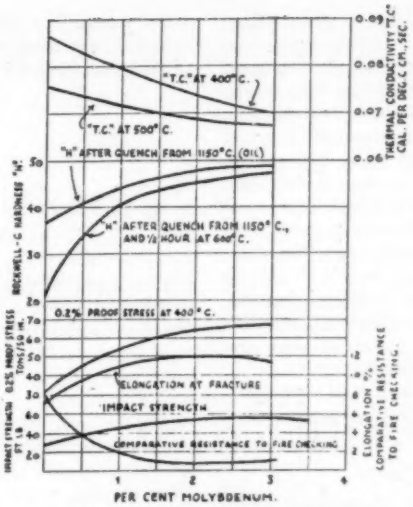

"T.C." AT 400°C.
"T.C." AT 500°C.
"H" AFTER QUENCH FROM 1150°C. (OIL)
"H" AFTER QUENCH FROM 1150°C., AND ½ HOUR AT 600°C.
0.2% PROOF STRESS AT 400°C.
ELONGATION AT FRACTURE
IMPACT STRENGTH
COMPARATIVE RESISTANCE TO FIRE CHECKING
PER CENT MOLYBDENUM.

Fig. 2.—Influence of molybdenum on the properties of 0·25/0·30 C—1·5 Cr steel.

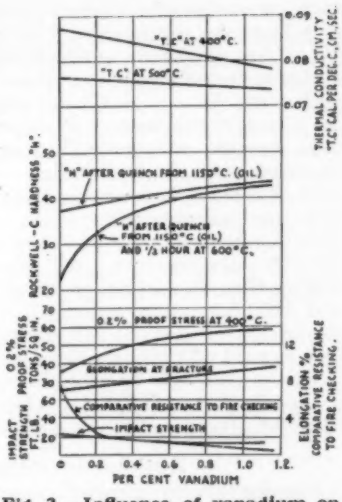

"T.C." AT 400°C.
"T.C." AT 500°C.
"H" AFTER QUENCH FROM 1150°C. (OIL)
"H" AFTER QUENCH FROM 1150°C (OIL) AND ½ HOUR AT 600°C.
0.2% PROOF STRESS AT 400°C.
ELONGATION AT FRACTURE
COMPARATIVE RESISTANCE TO FIRE CHECKING.
IMPACT STRENGTH
PER CENT VANADIUM

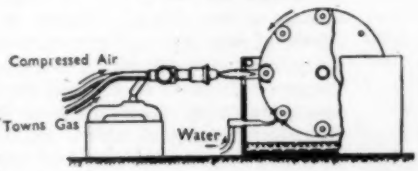
Fig. 3.—Influence of vanadium on the properties of 0·25/0·30 C—1·5 Cr steel.

A summary of the findings of the investigation as a whole is given in Fig. 6 and in Table II, where are listed the salient properties of the various groups of

TABLE II.

Steel Group No.	W, %	Mo, %	V, %	0·2% Proof stress at 400° C. Tons/sq. in.	Resistance to fire checking at 600–650° C.	Thermal conductivity at 400° C. cal/°C., cm., sec.
4a.	3	0·5–3·0	—	65·4	Good	0·070
4b.	5	0·5–3·0	—	71·8	Very good	0·069
5a.	3	—	0·2–1·0	69·2	Still sufficient	0·072
5b.	5	—	0·2–1·0	69·9	Good	0·069
6a.	—	0·5	0·2–1·0	69·9	Sufficient	0·079
6b.	—	1·0	0·2–1·0	75·6	Rather small	0·07ₒ
7a.	3	1·0	0·2–1·0	72·4	Good	0·073
7b.	3	2·0	0·2–1·0	73·7	Good	0·072
7c.	3	3·0	0·2–1·0	62·2	Good	0·071
8a.	5	1·0	0·2–1·0	73·0	Mostly very good	0·069
8b.	5	2·0	0·2–1·0	69·2	" " "	0·069
8c.	5	3·0	0·2–1·0	55·9	" " "	0·069

Fig. 4.—Device for testing the resistance to fire checking.



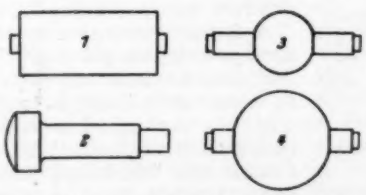


Fig 5.—Test specimen used in device shown above.

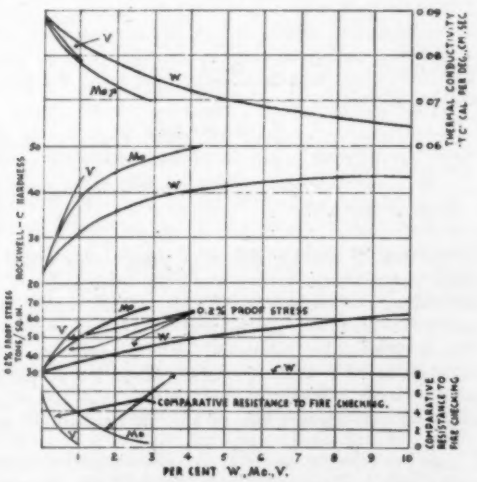


Fig. 6.—Comparison of the respective influences of tungsten, molybdenum and vanadium upon the properties of 0·25/0·30 C—1·5 Cr steel.

steels investigated. These results show once more that it is the tungsten content which is responsible for a high resistance to fire checking; and it is also seen that from this aspect neither vanadium nor molybdenum can be considered as satisfactory substitutes. As regards the influence of alloying constituents upon the strength at elevated temperature, the most efficacious element is vanadium, while tungsten is the least efficacious, the remedy lying of course, in the use of higher tungsten percentages. As was pointed out above, tungsten possesses the advantage that its adverse influence upon the thermal conductivity is smaller than that of either vanadium or molybdenum; and this fact compensates to a certain extent the diminution in thermal conductivity incurred with the employment of high tungsten percentages.

The conclusion to be drawn is that tungsten represents an invaluable and indispensable alloying constituent of tool steel to be used for hot work.

Russian Investigation

Russian efforts directed towards dispensing with tungsten as alloying element in tool steels for hot work appear to have been mainly directed towards the employment of silicon-manganese-chromium materials, although an 8% tungsten steel and an 8% Cr–3% W steel were also much used at one time.

Unlike the German investigation referred to above, the Russian work under review was concerned with ascertaining the service life of hot work punches and dies under actual operating conditions.* The compositions investigated were the following:

I. ..	0·46 C.	3·1 Si	0·45 Mn.	8·18 Cr.	
II. ..	0·54 C.	0·38 Si	1·39 Mn.	0·72 Cr.	0·28 Mo.
III. ..	0·35 C.	1·4 Si.	1·05 Mn.	1·3 Cr.	
IV. ..	0·64 C.	0·3 Si	0·7 Mn.		

The plain carbon steel No. IV was included solely for the purpose of comparison. The tool life tests were carried out with the use of water-cooled punches, the work piece being of structural carbon steel at 1,140–1,160° C. temperature, the punching process having a duration of from five to eight seconds.

The tools in the tempered condition had a troostitic structure, except those made of No. III steel, which was tempered at 650° C., and showed a sobitic structure. For each test series some 30–40 punches of the same material and of identical heat-treatment were employed, care being taken to maintain constant test conditions. The following heat-treatment was used for the various steels, to wit:

TABLE III.

			Hardness Brinell.
Steel: I ..	1,050° C.	Tempered at: 650° C. ..	364–387
II ..	825° C.	.. 540° C. ..	364–418
III ..	930° C. Quench in oil to 250° C., followed by air cooling	.. 540° C. ..	364–444
III ..	930° C.	650° C. ..	286–302
IV ..	850° C.	380° C. ..	340–380

The useful service life of a punch was considered to have terminated when working had led to erosions exceeding 0·4 mm. in depth. The tools made of steels II and III developed scattered erosion marks which gradually increased during service life. The carbon steel IV was, of course, subjected to particularly severe wear. Taking steel No. II as standard, the comparative service life of the various steels was:

No. II	100%
No. III	200–220% (650° C. temperature).
No. I	100–120%
No. IV	30–40%

* J. A. Heller and A. Y. Truskov, *Stal'*, No. 5/6, 1945, p. 45–48.

Of particular interest are the respective hardness values of the steel after tempering and after completion of their service life, as given below:

HARDNESS BRINELL.

	New tool	Discarded tool	Critical point Ac_1, °C.
II	364–418	444–550	790
III—Tempered at 540° C. ..	418–444	196–241	860
III—Tempered at 650° C. ..	286–302	196–241	860
I	364–387	196–241	975
IV	340–380	196–241	760

This table shows that steel II is the only one which exhibits an increase in hardness during the tool life, the case extending to a depth of 1·5–2·0 mm. This steel was found to have fine-grained martensitic structure, while the punches made of steel No. I and steel No. III had a fine-grained pearlitic structure. Both the microstructure and the change in hardness indicated that the surface of the die must have reached some 800°–850° C. In the case of steel II and IV this is higher than the Ac_1 point, while it is still below the Ac_1 point of steels Nos. I and III. It is obvious that in the case of No. II steel the cooling power of the internal water-cooling system must have been sufficient to quench this material.

The thermal conductivity of No. II steel in the tempered condition was found as 0·32 cal/cm. °C., and that of No. I steel was only 0·17 cal/cm. °C. These data are stated to be well in line with the precision measurements made by the other Russian investigators with the employment of the Kohlrausch method. Comparative measurements of the four steels were also carried out by measuring their electrical conductivity, which, according to Wiedemann and Franz, is a function of the thermal conductivity. The data obtained served as confirmation of the fact that the steel No. I has the lowest thermal conductivity of the four steels considered, the plain carbon steel No. IV having the highest conductivity. Owing to the low thermal conductivity of steel No. I the removal of heat from the working surface of the punch is considerably retarded, which must result in a higher surface temperature than encountered with the other steels investigated. This fact was fully confirmed by the surface colour of the discarded tools.

The importance of the influence of the mechanical properties of the steel upon the service life of the punching tools was demonstrated by steel No. III, insofar as this material yielded by far the best results. Steels of this type are known to exhibit a considerable drop in impact strength when passing the temperature range of 530–570° C. heating for tempering; while at the same time their hardness increases to 364–440 Brinell. Heating to 650° C. raises the impact strength to 10–12 mkg./cm.², with a corresponding fall in hardness to 280–300 Brinell. In view of this peculiarity dies made of No. III steel were heat-treated in two different ways, as indicated in Table III.

A further conclusion to be drawn from the breakdown test is that the tool life is little dependent upon the initial hardness and impact strength. The decrease in hardness suffered in service does not materially affect the mechanical strength of the tool, since the loss in hardness is confined to the surface stratum. In fact, it was found that punches broke down quickly when the steel was in heat-treated condition, and had high hardness and correspondingly low impact strength,

failure being due to the appearance of long, deep cracks. On the basis of the tool life of the carbon steel No. IV, the influence of the heat-treatment of steel No. II was as follows :

Carbon steel No. 4	100%
Steel No. III, tempered at 540° C.	30–50%
Steel No. III, tempered at 640–670° C.	200–250%

Conclusions

The results of these tests show that good thermal conductivity of the steel and a high Ac_3 point are factors of importance. Chromium and tungsten tend to raise the critical point, but at the same time, they also diminish the thermal conductivity of the steel. In the opinion of the Russian metallurgists, large percentages of tungsten are not therefore always to be recommended. It is stated that the required mechanical strength can be achieved by adding 1–1·5% chromium, while the required high critical temperature can be achieved by a silicon content of 1·0–1·5%. The carbon content should be kept at 0·3–0·4%, as higher carbon percentages in combination with increased silicon and chromium contents impair the thermal conductivity, besides depressing the critical Ac_3 point. Russian metallurgists suggest that punches for hot work should preferably be made of a material containing 0·3–0·4% C, 1·0–1·5% Cr, and 1·0–1·5% Si, and they claim that these specifications are met by the aforementioned steel No. III.

Abuse of Patent Monopoly Rights

By S. T. Madeley

The abuse of monopoly rights as regards patents is discussed in the light of the Second Interim Report issued by the Departmental Committee appointed to consider amendment of the Patent Acts. The report is summarised, attention being directed to legal proceedings in relation to patents and that much discussed question—inventive merit.

A SECOND Interim Report has been issued by the Departmental Committee appointed by the Board of Trade to consider amendment of the Patent Acts, and very interesting reading it makes, from many points of view. It deals more particularly with abuse of monopoly rights as regards patents, with legal proceedings in relation to patents, and that much-discussed question—inventive merit.

The Report at the start points out that the Soviet Union is the only important country that has an alternative method to that of the patent system for rewarding inventors, and at the same time notes that this other method is bound up with the economic system of that country and that but little is known as to how it actually functions in practice. There is agreement with the conclusions reached by the American National Patent Planning Commission that the Patent System should be retained, although it is capable of improvement.

After giving a résumé of the history of the Patent Act as it now stands, the allegations relating to restrictive use of patent rights are dealt with. Mainly they are that the patentee on occasion fails to use the invention at all, though others want it ; that only part of the potential market for the commodity is supplied ; that technical development is hampered by the refusal of the patentee to grant licences under a patent covering an important new process ; that through patents a greater monopoly is built up than that conferred by the patent rights ; that as a condition of using a patented process restrictive conditions are attached to licences ; that industry is controlled by groups of firms having licensing and patent-pooling arrangements ; and that foreign firms use British patents to prevent production in this country and keep the British market for themselves. These allegations are put forward as possibilities of course.

On the whole the Committee finds these allegations exaggerated, although it is alive to the fact that abuses of some of the kinds indicated do occur. According to the Committee Section 27 (Abuse of Monopoly) should be strengthened, for example to cover practices which fall short of malpractices. It is pointed out that abuses still occur, although but few applications for relief have been made. As regards the " Licence of Right " Section 24, but few are licences granted under this section. When there is grant it is usual for the indorsement to be cancelled. Consequently the section seems mostly to be used for saving money on doubtful patents. In practice it has been found that Section 38 has been so drawn that it can be easily avoided so that a licensee may have to accept a licence on terms which are restrictive. Only one effective application has been made for a licence under Section 38 A (3) which concerns patents relating to food or medicine. This state of affairs has been put down to fear of retaliation, to uncertainty as to what the Comptroller will decide, and to the trade mark situation.

The Committee has considered various suggested remedies for the abuses and so on complained of.

There was a proposal to indorse all patents " Licences of Right." It was feared that this would discourage patenting and encourage secrecy. Development and research would be discouraged. Modifications of this idea were also turned down for various reasons. Similarly a proposal to extend the scope of Section 38 A (3) to cover all patents was rejected.

There should be more facilities for granting compulsory licences. For instance, an applicant should be able to apply where he can open up a new field of manufacture, or supply a neglected or insufficiently supplied export market. Similarly, where a patent would hinder an inventor who has substantially contributed to the art and cannot get a licence on reasonable terms.

In considering an application for a licence under the section the Comptroller should be guided by several principles, namely, to secure the fullest possible working of the invention here and for the export market consistent with the fair treatment of those already engaged in working or developing the invention under the British patent ; to remunerate reasonably those beneficially entitled to the patent taking the nature of the

invention into consideration ; to make available to the public at the lowest possible price food, medicine, and curative or surgical devices ; the working of the invention by the applicant to the public advantage ; the use of the invention made by the patentee and licensees ; the risks which the aspiring licer see would run in financing and working the invention should he obtain the grant. All applications should be determined in view of the conditions present when the application is made. It should not be permissible for a licensee to add restrictive conditions relating to purchase of a patented article when granting a sub-licence.

In Section 38 (1) it should be required that the terms of the option which is referred to therein should be fully set out. When application is made for relief under Section 27 the applicant should not be prejudiced by having availed himself of the terms offered him by the patentee. This should be made clear in Section 38 (1).

The person from whom a patent or interest therein is acquired should be entitled to register the deal in the Patent's Register.

For a long time it has been a matter for acute discussion between patentees and inventors whether they would be better off or not if the Comptroller were empowered to consider subject-matter during the passage of an invention through the Office. At present the Comptroller must grant patents in cases where there is no inventive merit and involving no more than the application of elementary common knowledge, although he can direct the applicant to insert in his specification a reference to an earlier one as a warning to the public.

An obviously invalid patent may serve as a deterrent to manufacturers because of the expense incurred in patent litigation even where successful. Trifling improvements in or modifications of a main patented invention may in turn be patented and in effect serve to prolong the life of the original patent. Also a licensee under the main patent may have to take out licences under the subsidiary patents and agree not to question their validity.

The Committee is satisfied that there would be little likelihood of the cost of patenting being greatly increased or of valuable inventions being wrongfully rejected by the examining staff of the Patent Office, if the Comptroller were to consider subject matter.

After pointing out that the Patent Offices of the principal industrial countries such as the United States of America, Germany, Holland, and Sweden do consider subject-matter without apparent harm to the interests of inventors or public, but indeed within the opinion of some people with enhanced value to the patents, the Committee proposes that the Comptroller should be given power to reject applications where the inventions in question are devoid of subject matter. To begin with, until standards are established as regards inventive merit, there should be a hearing before two Hearing Officers sitting together and rejection should only follow on a unanimous decision of the Hearing Officers.

Section 75 should be amended to enable the Comptroller to refuse obviously trivial cases. Under Section 7 he should be empowered to refuse cases in which the applicant either cannot or will not clearly set out precisely what he has added to prior knowledge. Lack of subject matter should also form a ground for opposition under Section 11 or for revocation under Section 26 of the Acts.

Prior use should not form a ground for rejection under Section 7 but should do so under Section 11 or 26. In the latter cases too interested parties can produce arguments in support of their views, in the former they cannot.

In the opinion of the Committee utility cannot be with convenience investigated or adjudicated upon by the Comptroller, except as regards furnishing samples under Section 2 (5) in connection with chemical cases. In revocation cases under Section 26 there should be an appeal to the Patents Appeal Tribunal with a further limited right of appeal to the Court of Appeal.

There is a general opinion that the judges who try patent actions are not sufficiently well equipped from the scientific and technical standpoints, however well versed in the law they may be. Two judges should be appointed to try patent actions who have suitable scientific or technical qualifications, besides those of a legal nature and experience of patent litigation. Either of the judges should be competent to preside over the Patents Appeal Tribunal. In appeals in infringement and revocation cases, that one of the two judges who has not heard the case should be competent to sit on the Court of Appeal. Leave to appeal to the House of Lords should only be given in important cases where there is reasonable room for doubt. A scientific assistant should sit with the special judge or judges unless his services can be dispensed with, and similarly when appeals are heard. Judges should make themselves acquainted with a case before hearing it. Expert witnesses should be permitted to inform the Court what they understand the inventor to have disclosed in their specifications.

If a party desires to submit experimental proof of his case he should have to supply to the opposite side full and precise particulars of the proposed experiments and what facts he claims thereby to establish. He should be informed if the demonstration is desired or not. At the trial of the action, the judge, on being satisfied that the facts are proved and relevant, should direct that the party requiring the demonstration should bear the cost thereof. There should be no further delivery of statements signed by counsel setting out the contentions on which the parties intend respectively to rely. All the appeals from the Comptroller's decisions should be brought before the Patents Appeal Tribunal on notice of appeal. The Comptroller should be empowered to decide questions of infringement provided both parties agree to accept the decision of the Comptroller as final and do not desire an injunction. Damages assessed under these conditions should not exceed a thousand pounds.

The number of British patents granted during the period 1934-1938 relating to annealing, tempering, and otherwise modifying the physical characteristics of metals is about 400. No doubt some of them will benefit by the above changes in the law when they come into force.

THE Drayton Regulator and Instrument Co. Ltd. has issued its first post-war publication in the form of a summarised catalogue. It gives the present manufacturing range of this Company and contains a considerable amount of useful information which can be used as a guide in the selection of suitable equipment for a given application. Copies are available on application to the Company at West Drayton, Middlesex.

Platinum-Iridium Alloys

By O. E. Zvyagintsev, A. G. Rakhshtadt and M. A. Vladimirova

The process of diffusion in platinum-iridium alloys has been studied with alloys containing 10, 25, 35 and 50% *iridium, prepared in the form of powder, and in view of the importance of forming solid solutions of these alloys at relatively low temperatures by the powder metallurgy method, it is thought that a translation of the report* of the work will be useful to many readers.*

INVESTIGATIONS which were carried out many years ago of the electrical conductivity[1], thermo-electromotive force[2], thermal conductivity[3] and density[4] of platinum-iridium alloys appeared to establish that these alloys, over a fairly wide concentration range, were homogeneous in their structure. With these studies must be associated the important work of Nemilov[5] (1929) which led to the conclusion, based upon extremely accurate determinations of the temperature coefficient of electrical resistance, combined with careful observations of the microstructure and the hardness, that there exists for these alloys an unbroken series of solid solutions over the whole range of concentrations. This point of view is fully in accordance with the behaviour of the temperature coefficient of the electrical resistance and the hardness. In the following year (1930), Feussner and Müller[6] established the liquidus line of the system to an accuracy of ± 5°. (The position of the solidus line is still somewhat hypothetical.)

Soon afterwards, Weerts[7] by means of X-ray analysis, showed that with platinum-iridium alloys—as also with platinum-gold, platinum-rhodium and palladium-gold alloys—the lattice period changes linearly with the concentration, a conclusion which confirms the results of the previous investigations on the existence of an unbroken series of solid solutions. In 1940, Masing's work appeared[8]. This disclosed a peculiarity, which had not been noticed before, in connection with the electrical resistance of alloys, containing up to 40% iridium, after heat-treatment at low temperatures. The resistivity is lowered. Masing also observed a very interesting relationship between the ultimate tensile strength and the heat-treatment temperature. (Incidentally, Masing's figures for tensile strength are surprisingly low.) The observed behaviour is indicative, according to Masing, of transformation in the solid solution.

The question of the possibility of transformations in an alloy of platinum-iridium was first considered by Le Chatelier[9] (1886) who found that heat was absorbed by an alloy of platinum with 20% iridium, at red-heat. To explain this effect, Le Chatelier postulated an allotropic transformation in the alloy. Subsequently Geibel[2] discovered that the ultimate tensile strength of cold-worked platinum-iridium alloys containing more than 10% iridium was not merely unreduced by heat-treatment at 750° but that it was actually increased and only showed a fall when very high temperatures were used. This fact was thought by Geibel to be due to a transformation in the iridium. Later on, Nowack[10], in reviewing Geibel's work, was led to the conclusion that platinum-iridium alloys containing 15–25% iridium evidence the phenomenon of dispersion hardening which enabled him to explain the increase in the tensile strength.

Masing, in the light of the above-mentioned facts, suggested that the formation of a superstructure might occur in these alloys. This idea of Masing's would appear quite probable. His results give a relationship between resistivity and composition which indicates the possibility of the existence of an ordered arrangement when the iridium content is 25%, corresponding with a superstructure of the type A_3B (Pt_3Ir.) Masing attempted to decide the presence of the superstructure by means of X-rays, but in this he was unsuccessful by reason of the fact that the dispersive powers of the atoms of platinum and iridium are extremely close.

Platinum-iridium alloys are distinguished from the other alloys and metals of the platinum group by their superior strength. As can be seen from Table I., compiled from the data given by Wise and Eash[11] for alloys in the annealed state, the strength increases proportionally with the iridium content.

TABLE I.
MECHANICAL PROPERTIES OF ANNEALED PLATINUM-IRIDIUM ALLOYS.

Composition	Elastic limit (kg./mm.²)	Ult. tensile strength (kg./mm.²)	Elongation on 50 mm. %	Reduction in area %
95% Pt+ 5% Ir	16·17–12·65	29·17–27·42	22–32	95–94
90% Pt+10% Ir	25·31–21·09	38·66–37·26	25–27	95–94
80% Pt+20% Ir	43·59–41·48	71·70–65·38	21–20	89–88

Platinum-iridium alloys, notwithstanding their high tenacity, have a fair degree of plasticity and are able to withstand a considerable amount of cold deformation resulting in improved mechanical properties[11 12]—vide Table II. The high strength figures are accompanied by unusually high values for the reduction in area.

Along with these high mechanical qualities, platinum-iridium alloys possess an extremely high degree of resistance to corrosion by many chemical reagents. This resistance increases as the iridium content rises. Platinum-iridium alloys containing more than 30%

* "A Study of the Mutual Diffusion of Platinum and Iridium in the Solid State." *J. App. Chem.* (U.S.S.R.), 1944, 17, 22–30.

1 W. Guertler, *Z. anorg. Chem.*, 1906, 51, 427; *ibid.*, 1907, 54, 73.
2 W. Geibel, *Z. anorg. Chem.*, 1911, 70, 246.
3 T. Barrat and R. M. Winter, *Ann. Physik.*, 1925, 77, 5
4 Ste. Claire-Deville and Debray, *Compt. Rend.*, 1875, 81, 839.
5 V. A. Nemilov, *Izvest. Inst. Platiny*, 1929, 7, 13.
6 O. Feussner and L. Müller, *Heraeus Festschrift*, Heraeus G.M.B.H., Hanau, 1930, pp. 14–15.
7 J. Weerts, *Z. Metallkunde*, 1932, 24, 839.
8 G. Masing, *Z. Metallkunde*, 1940, 32, 122.
9 Le Chatelier, *Bull. Soc. Chim.*, 1886, 45, 482.
10 L. Nowack, *Z. Metallkunde*, 1930, 22, 94.
11 E. M. Wise and J. T. Eash, *Trans. Amer. Inst. Min. Met. Eng.*, 1935, 117, 313.
12 F. Korn, *Metal Ind.* (Lond.), 1931, 38, 309.

TABLE II.
MECHANICAL PROPERTIES OF COLD-WORKED PLATINUM-IRIDIUM ALLOYS.

Composition	Elastic limit (kg./mm.²)	Ult. tensile strength (kg./mm.²)	Elongation on 50 mm. %	Reduction in area %
95% Pt+ 5% Ir	37·61	48·58	2·0	92
90% Pt+10% Ir	37·96	57·80	2·5	94
80% Pt+20% Ir	70·93	98·78	2·5	84·5
75% Pt+25% Ir	—	121·0	—	—
70% Pt+30% Ir	—	150·0	—	—

iridium are almost insoluble in hot, undiluted, aqua regia or, indeed, in any other mixture of hydrochloric and nitric acids. A comparison of the properties of metals and alloys of the platinum group in respect of resistance to corrosion is given in Table elsewhere[13].

Platinum-iridium alloys are widely used in a whole series of industries. Up to the present it has not been found possible to employ substitutes for them except on a very limited scale, whilst in such industries as, for example, those concerned with the construction of electric machines, platinum-iridium alloys are used exclusively because of their high resistance to the action of electric charges.

Production of Alloys

Platinum-iridium alloys are obtained usually by metallurgical means which include fusion and subsequently hot mechanical treatment. In the castings there remain gaseous bubbles, non-metallic inclusions, material derived from the crucible, etc. The gaseous bubbles cause the metal to flake during rolling into sheets and stamping operations. The presence of flakes and non-metallic inclusions is absolutely inadmissible in a large number of cases and because of this it has been suggested by O. E. Zvyagintsev that the method of powder metallurgy—metallo-ceramics—should be employed for the manufacture.

Powder Metallurgy Methods

Metallo-ceramics was first used for platinum at the end of the 18th century. Afterwards, the method was extensively used in the preparation of such difficultly fusible metals as tungsten, molybdenum, tantalum and niobium. Nevertheless, the metallo-ceramic method of obtaining platinum had to be given up and replaced by melting in furnaces since the latter method, on account of the additional refining could yield a purer platinum. At present, the metallo-ceramic method of obtaining platinum which was described some time ago by Wollaston[14], is again assuming great importance in as much as the refining method which is employed guarantees a high degree of purity of the platinum and in this case melting is no longer a purifying operation but even a means by which impurities are introduced. Moreover, the loss of an expensive metal such as platinum is reduced.

Work which has already been carried out in this direction has indicated that the metallo-ceramic method of obtaining platinum is quite practicable and advantageous. The broad outlines of the metallo-ceramic method as applicable to pure platinum are described in the literature[15 16] but no account of any kind is given of the preparation of alloys of platinum.

Certain difficulties appeared at the start when the method was considered in connection with platinum alloys. The preparation of alloys by means of metallo-ceramics requires that there shall be considerable diffusion in the solid state between the components of the alloy which is to be produced. Information obtainable from the literature tends to show that the speed of diffusion in these alloys cannot be very high. For example, Nemilov gives evidence by means of photomicrographs that considerable chemical non-homogeneity exists after heat treatment at 1,100° C. for 10 days.

Notwithstanding the unfavourable prospect created by these data it was decided to undertake a detailed study of diffusion in these alloys. The investigation proved to be extremely interesting and practically important.

If reference be made to van Liempt's[17] experiments with the alloys Au-Pt, Au-Pd and Au-Ni one finds that the coefficients of diffusion of gold in platinum, palladium and nickel are all the same whereas the coefficients of diffusion of platinum, palladium and nickel in gold are all different. This experimental fact is extraordinarily interesting for, according to Mehl[18], the opposite effect would not have have been unexpected. It has been established also that, with most of these alloys, the diffusion speed is inversely proportional to the temperature at which the metal melts. This rule is by no means generally applicable as was pointed out by Mehl[18 19]. (See Table III.)

TABLE III.
DIFFUSION IN PRECIOUS METALS (AFTER MEHL[18])
Solvent metal Ag Atom. rad. = 1·442, M.P. = 960·5° C.
Au Atom. rad. = 1·438, M.P. = 1,063° C.

Dissolving metal	Coefficient of diffusion cm.²/sec. at 900°	Q	Atomic radius (Goldschmidt)	Melting point °C.
Au	$1·6 \times 10^{-9}$	29,800	1·438	1,063
Alloy Ag-Au	$1·6 \times 10^{-8}$	26,000	—	—
Pd	—	—	1·372	1,555
Alloy Ag-Pd (20% Pd)	$1·1 \times 10^{-9}$	10,200	—	—
Cu	$9·0 \times 10^{-9}$	27,400	1·276	1,083
Alloy Au-Cu (10% Cu)	$5·0 \times 10^{-9}$	—	—	—
Ni	$0·34 \times 10^{-9}$ rich $6·0 \times 10^{-10}$ rich	—	1·244	1,452
Pd	$0·34 \times 10^{-10}$ rich $6·0 \times 10^{-10}$ rich	—	1·372	1,555
Alloy Au-Pd	$1·0 \times 10^{-10}$	37,300	—	—
Pt	$0·14 \times 10^{-10}$ rich $0·61 \times 10^{-10}$ rich	—	1·385	1,755
Alloy Au-Pt	$0·77 \times 10^{-10}$	39,000	—	—

Experimental

The work was planned to cover a study of the diffusion of platinum and iridium, the metals being taken in definite relative proportions—90% Pt and 10% Ir, 75% Pt and 25% Ir, 65% Pt and 35% Ir and 50% Pt and 50% Ir. These ratios were to be obtained by mixing appropriate quantities of platinum and iridium in the form of powder.

Iridium was obtainable in the powder form from the refining works but the platinum was mostly in the form of ribbon which had to be transformed into the powder form by fusing with lead and then extracting the lead by means of nitric acid. After repeated extractions and

13 Steel, 1940.
14 W. H. Wollaston, *Phil. Trans. Roy. Soc.*, 1929, 8, 119.
15 R. H. Atkinson and A. R. Raper, *J. Inst. Metals*, 1936, 59, 179.
16 O. E. Zvyagintsev, Jubilee vol. of the scientific work of the Moscow Inst. of Non-Ferrous metals and Gold, 1930–1940.
17 van Liempt, *Rec. Trav. Chim.*, 1932, 51, 114.
18 R. F. Mehl, *J. Appl. Physics*, 1937, 174.
19 R. F. Mehl, *Trans. Amer. Inst. Min. Met. Eng.*, 1936, 122, 11.

ignitions the powder was found to consist of grains with diameters: 0–1 μ (62%), 1–2 μ (37%) and 2–3 μ (1%). The iridium powder was also very fine: 0–1 μ (59·5%), 1–2 μ (37%) and 2–3 μ (3·5%). Spectrographic analysis of the mixed powders indicated that iron and rhodium were absent whilst gold and lead were present in amounts less than 0·1%. The presence of lead was obviously due to the method used for preparing the platinum powder. It was not detected by chemical means in the platinum.

The study of diffusion was carried out by means of electrical resistance measurements, by micrographic and by X-ray methods. Resistance measurements were made by Kelvin's Double Bridge method. Micrographic observations were made several times in the course of the diffusion process. For etching the polished specimens chlorine was used. This was obtained by heating a mixture of potassium bisulphate, manganese dioxide and common salt. A Debye camera was employed in the X-ray investigations and the phasal composition and the parameter of the phases were determined. The tube (Müller) had a copper anti-cathode and was rated at 8 milliamps, 50 kw. The exposure time was usually nine hours.

Sequence of Experiments and Working Conditions

The weights of platinum and iridium powders required to give mixtures of the desired composition were mixed in a ball mill containing platinum-iridium alloy balls. The mixed powders were then compressed in a special form of press under a pressure of 6,700 kg. per sq. cm. The samples so obtained had dimensions 0·4 × 3 × 15 mm. and were used for specific electrical resistance and microstructure. Unfortunately, we were obliged to give up the idea of determining the temperature coefficient of electrical resistance in this instance since the heating brought about diffusion and also a change of density, it would have been difficult to distinguish the influences of these processes. For the purpose of X-ray analysis small cylinders (diameter about 0·2 mm.) were prepared from the above-mentioned samples.

The diffusion process was studied in detail at the temperatures 1,300° and 1,400° but at 1,500° the examination was confined to X-ray analysis and the microstructure. The duration of the heating was 40 hours at 1,300°, 10 hours at 1,400° and 5 hours at 1,500°. A vertical electric furnace with resistance elements of alloy No. 2 was used for the tests at 1,300° and 1,400° whilst the heating at 1,500° was carried out in a fused quartz tube inserted in a furnace with molybdenum resistance elements. In all cases the temperature was controlled by means of a platinum-rhodium thermo-couple.

Results of Measurements of Specific Electrical Resistance

Resistivity results are given in Table IV. From these it is clear that at 1,300°, with each of the compositions studied, as the duration of heating is increased the specific electrical resistance becomes greater and attains a maximum after 10 hours at this temperature. There is then a gradual fall. The actual value of the maximum attained is greater, the greater the iridium content of the alloy. The behaviour at 1,400° is somewhat similar but the maximum is lower and appears after a shorter time of heating. A theoretical interpretation of these results is difficult by reason of the fact that during the heating there takes place both diffusion processes and shrinkages, having opposite effects upon the specific resistance. If the diffusion process, by which a solid solution is formed, leads to an increase of the specific electrical resistance, then the condensation process leads to a decrease. Nevertheless, one may remark with reference to the heating temperature of 1,300° that the predominating process in periods up to 10 hours is diffusion. With prolonged heating the condensation process has a greater effect. Similar characteristics are observable at 1,400° but in this case the speeds of both processes are noticeably greater than they are at 1,300°.

TABLE IV.

Change of resistivity $\left(\frac{\text{ohms. mm.}^2}{\text{m.}}\right)$ during heating—mean values.

Composition	Temp.	Duration of Heating (hours).								
		0·5	1	3	5	10	15	20	25	40
90%Pt+10%Ir	1,300°	—	0·251	0·312	—	0·521	—	0·400	0·394	0·396
75%Pt+25%Ir		—	0·365	0·500	—	0·708	—	0·686	0·642	0·626
65%Pt+35%Ir		—	0·416	0·545	—	0·781	—	0·759	0·735	0·753
50%Pt+50%Ir		—	0·416	0·515	—	0·892	—	0·780	0·774	0·760
90%Pt+10%Ir	1,400°	—	0·252	0·376	0·380	0·371	0·373	—	—	—
75%Pt+25%Ir		0·402	0·415	0·542	0·550	0·536	0·533	—	—	—
65%Pt+35%Ir		0·460	0·483	0·547	0·568	0·521	0·514	—	—	—
50%Pt+50%Ir		0·426	0·590	0·600	0·615	0·552	0·546	—	—	—

Metallographic Analysis

The alloys examined for microstructure were the same as those which were studied in respect of electrical resistance. After grinding and polishing all the samples investigated were etched by means of a mixture of potassium bisulphate and sodium chloride to which manganese dioxide was added. The etching agent was the chlorine produced as a result of the reactions of these substances. From the microphotographs (not reproduced in the original paper for reasons outside the control of the authors of the paper and the editors of the journal) it is possible to trace the kinetics of the building up of the structure and the formation of the solid solution. After one hour's heating at 1,300° (of the 10% iridium alloy) there is no indication of structure, but after 5 hours grains of a solid solution are present and after 10 hours these are obviously characteristic of a solid solution with a polyhedral structure. At 1,400° the picture is similar but a completely homogeneous state is attained after only 5 hours' heating. At 1,500° the diffusion process which produces the solid solution takes place still more rapidly and a homogeneous solid solution is obtained in 3 hours. Similar changes of structure are observed in the alloy with 25% iridium. In this case as in the previous one a homogeneous solid solution is obtained after 10 hours' heating at 1,300°. At 1,400° 5 hours is sufficient and at 1,500° the process of obtaining a homogeneous and compact alloy is greatly accelerated. It has to be noted, however, that the majority of the samples submitted to the heating operation were not altogether compact and there remained pores which were visible on the microphotographs. The presence of pores caused the values for resistivity to be higher for such samples. Experiments which were carried out subsequently indicated that a completely compact metal could be obtained without altering the pressure used in the preparation of the blocks if vacuum heating were employed in the study of the diffusion process. The series of microphotographs which were taken of the alloys with 35% and 50% iridium at the temperatures 1,300°, 1,400° and 1,500° gave clear evidence of the formation of a homogeneous solid solution.

X-ray Analysis

The results of X-ray analysis were in full agreement with the micrographic and resistivity investigations. A fairly considerable difference in lattice periods between platinum (3·915 A) and iridium (3·831 A) permitted the formation of the solid solution to be followed clearly. It was noticed that when the micrographic evidence pointed to the presence of a solid solution, the X-ray evidence also indicated a single phase. In some cases, particularly when the iridium content was high, although the grains of the solid solution were already conspicuous on the microphotographs, the X-ray diagram indicated the presence of one phase but the interference lines remained somewhat blurred, showing that there was some non-homogeneity of the alloy. Further heating did in fact, lead to the obtaining of fairly sharp interference lines. At 1,300° a heating period of 20 hours was quite sufficient to obtain an X-ray picture of this kind. If the final values of the lattice parameters of the alloys investigated are plotted as a function of the concentration it is clear that a linear relationship is observed. These findings corroborate the earlier work of Weerts[7] and, of course, confirm the existence of an unbroken series of solid solutions in each of the cases studied. From this fact it is possible to determine, approximately, the composition of the phases at any moment of the process. In the alloy with 25% iridium there are present, after 1 hour's heating at 1,300°, two phases with lattice constants, a_1=3·915 A and a_2= 3·857 A. After 5 hours there are still two phases and the lattice constants are, a_1=3·907 A and a_2=3·870 A but after 10 hours' heating the alloy appears to be homogeneous.

If, by making use of the relationship between the lattice period and the concentration, one determines the compositions of the phases, possessing the lattice constants given above, then it appears that after one hour's heating there are present together in the alloy both platinum and a solid solution of platinum and iridium of the composition, 70% iridium and 30% platinum. After 5 hours' heating the two phases are (1) a solid solution of iridium in platinum—8% iridium and 92% platinum, (2) a solid solution of platinum in iridium—53% iridium and 47% platinum.

From the results referred to it is possible to obtain information about the amounts of the phases present at different stages of the process. A simple calculation indicates, in the case of the alloy with 25% iridium that after one hour's heating, the quantity of platinum (or of the phase rich in platinum) has fallen to 64·5%, the reduction being due to the passage of some of the platinum into the iridium, with the formation of a solid solution. After 5 hour's heating at 1,300° the platinum phase comprises 63·6%. It is obvious that a rapid increase of the quantity of the platinum phase must occur for after 10 hours' heating we have an alloy which is a platinum-based solid solution.

A consideration of the evidence of X-ray analysis, photomicrography and electrical measurements leads to the conclusion that solid solutions are formed over all the concentration ranges studied in the system platinum-iridium, although, judging from the parameter changes of the phases rich in iridium, solid solutions are also obtained with greater contents of iridium and thus they occur within the limits of each system. The excellent agreement in the results obtained by the various methods is especially noteworthy. For example, the maximum on the resistivity curves agrees in respect of time with the appearance of a structure indicative of a homogeneous solid solution and the presence, determined by X-ray analysis, of a single phase.

Summary and Conclusions

1. The process of diffusion in platinum-iridium alloys has been studied with alloys containing 10, 25, 35 and 50% iridium which were prepared from their components mixed in the form of powder. The process was studied at temperatures 1,300°, 1,400° and 1,500° over periods up to 40 hours.

2. It was found that the diffusion process takes place very intensively. In all the alloys considered a solid solution was present after only 10 hours' heating at 1,300°.

3. The formation of a homogeneous solid solution was indicated by the appearance of a maximum on the curves for specific electrical resistance and the time agreed with the evidence obtained from microphotographs and X-ray analysis.

4. The results of X-ray analysis indicate that the alloys studied obey Vegard's law—there is a linear relationship between the lattice parameters and the concentration.

5. From the X-ray data the conclusion is drawn that the rate of diffusion of iridium into platinum is lower than that of platinum into iridium. A similar effect is known to occur with many precious metals and it finds expression in the rule: the higher the melting point, the lower the diffusion rate.

6. The formation of solid solutions at comparatively low temperatures and in relatively short periods of time is of practical importance from the point of view of the possibility of employing the method of powder metallurgy for the technical preparation of platinum-iridium alloys. It is possible, even at 1,300°, to obtain a completely homogeneous alloy of platinum with 25% iridium, whilst in the usual metallurgical method it is necessary to work at a temperature above 1,900° which presents certain well-known difficulties. If, however, one manages to overcome these temperature and furnace difficulties in connection with the 25% iridium alloy, one meets still greater difficulties in attempting to make alloys containing, say 50% iridium, by metallurgical means. By applying the metallo-ceramic method one may simplify the operations, raise the purity and cut down wastage. In addition to all this, the method could be used for obtaining directly—avoiding casting, forging, rolling and other operations—articles with simple and most serviceable forms and dimensions.

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GAS CARBURISING

By Ivor Jenkins, M.Sc.

The carburisation of iron and steel in solid and in gaseous media is discussed. The process mainly used in this country is that of box- or pack-carburising, but liquid carburising agents are also used to a considerable extent. The use of gaseous media is of more recent origin, and has made considerable progress in America, probably because of the ready availability of industrial gas high in hydrocarbons. Very few publications deal with the carburising properties of pure and complex gas mixtures ; the present paper, however, gives the results of an investigation which has been carried out over a period of years at the Research Laboratories of the General Electric Co., Ltd., into the development of industrial carburising atmospheres, some of the main aspects from which are given in this article.*

THE carburisation of iron and steel may be effected by heating in a solid, liquid or gaseous carbon-containing medium, which at high temperatures provides a supply of nascent carbon for absorption by the material being carburised. By controlling the temperature and time of treatment, the concentration of carbon on the surface of the steel and the depth of penetration may be varied over wide limits. The process mainly used in this country has been that of box- or pack-carburising which, by modern standards of heat-treatment practice, has certain objectionable features. Among these is the general lack of control over the process, with regard both to the temperature of the packed work and to the basic reactions taking place in the box. In addition, the economic aspect has to be considered, which involves the storage and handling of large quantities of carburising compounds, and the cost of heating the carburising compound, and the heavy carburising boxes to the necessary temperature.

Liquid carburising agents are used industrially to a considerable extent, and are of more or less the same composition—namely, a 50 : 50 mixture of sodium cyanide and sodium carbonate. From the point of view of ease of operation, temperature control and cleanliness, the process is superior to pack-carburising. Nitriding, as well as the carburisation of the steel takes place, but the maximum surface carbon content which can be obtained economically does not exceed about 0·8%. On the whole, the process is not suitable for the treatment of massive parts or for the production of deep cases.

Carburisation in gas is by no means of recent origin, although industrial development of the process in this country has been slow. The publication of Giolitti's[5] book on the cementation of iron and steel indicated the possibilities of carburising in hydrocarbons diluted with inert gases to minimise soot-formation due to decomposition of the carburising gas. This principle has formed the basis of most of the subsequent development work on processes suitable for industrial application, but it is only in recent years, since the rapid growth of controlled-atmosphere processes, that any real advance has been made. In this respect, probably because of the ready availability of industrial gases high in hydrocarbons, American practice has progressed far beyond that at present obtaining elsewhere.

Apart from the work of Giolitti, and more recently that of Bramley and his co-workers,[7] who studied the cementation of iron in pure and carburetted carbon monoxide, there have been very few publications dealing with the carburising properties of pure and complex gas mixtures which are suitable for use on an industrial scale. The present paper gives the results of an investigation which has been carried out over a period of years at the Research Laboratories of the General Electric Co., Ltd., into the development of industrial carburising atmospheres.

The Mechanism of Carburisation

Preliminary consideration is given to the mechanism whereby carbon is transferred to steel in both the solid and the gaseous carburising processes.

Pack Carburising.—In solid carburising the reactions whereby carbon is transferred to the steel are essentially gaseous. The generally accepted view is that carburisation is primarily effected by carbon monoxide, generated by a reaction between the charcoal and the air occluded in the carburising container. During the early stages of heating, the gas formed is principally carbon dioxide, but at higher temperatures the concentration of the dioxide relative to the monoxide decreases in order to satisfy the equilibrium :

$$CO_2 + C \rightleftharpoons 2CO \qquad (1)$$

Carburisation of the steel then proceeds *via* the breakdown of the carbon monoxide, and may be represented by the equation :

$$2CO \rightleftharpoons C_{Fe} + CO_2 \qquad (2)$$

The carbon dioxide formed in reaction (2) combines with the charcoal to form more monoxide, and thus the cycle of reactions is completed. The main function of the carbonate energiser is to provide a supply of carbon dioxide by slow dissociation at the carburising temperature, and thereby increase the concentration of carbon monoxide in the box. Oxide energisers function in a similar way, by forming carbonates with the carbon dioxide at low temperatures, and later evolving the gas at the carburising temperature.

Gases in a Carburising Box. A complete understanding of the various reactions involved in the pack-carburising process is important in that it may have a bearing on the development of atmospheres suitable for carburising. One fact which has emerged from the various investigations on the subject is that the process

* Communication from the Staff of the Research Laboratories of the General Electric Co., Ltd., to the Iron and Steel Institute. Advance copy, June, 1946. Written discussion should be addressed to the Office of the Institute, 4, Grosvenor Gardens, London, S.W. 1, to be delivered not later than November 2, 1946.

5 F. Giolitti, "Cementation of Iron and Steel." New York, 1915. McGraw-Hill Book Co., Inc.

7 A. Bramley and A. J. Jinkings. *The Iron and Steel Institute*, 1926, *Carnegie Scholarship Memoirs*. Vol. 15, pp. 17, 127.

of carburising by carbon monoxide is speeded up considerably in the presence of solid carbon. However, the effect of gases other than carbon monoxide must be considered in box-carburising. Water vapour absorbed in the charcoal of the carburising compound appears to influence considerably the composition of the atmosphere inside the container. Experiments carried out to obtain analyses of gases in contact with specimens in the process of carburisation gave the following results :

COMPOSITION OF GAS EXTRACTED FROM CARBURISING BOX.
Carburising temperature : 900° C. Steel : 3% nickel case-hardening steel.
Carburising compound : 80% oakwood charcoal—20% barium carbonate.

Test No.	CO_2, %	CO, %	H_2, %	Ratio CO_2/CO
1	0·8	30·6	14·9	0·026
2	0·9	35·6	16·2	0·025
3	1·2	32·3	17·0	0·037

The concentration ratio of the CO_2 and CO in the gases extracted tends to fall between the equilibrium values for saturated austenite and solid carbon, and, unless the excess of CO_2 is removed from the proximity of the steel surface, carburisation by the CO alone to give saturated austenite is not possible. The other feature of the analysis is the high concentration of hydrogen in all the gas samples extracted. It is considered that the hydrogen formed plays an important role in the process, either by cleaning up absorbed films of CO_2, or by the cyclic formation of hydrocarbons by direct reduction either with the charcoal or with the oxides of carbon. That hydrogen in the presence of carbon monoxide increases the rate of carburisation has been demonstrated by Bramley.[7].

Gas Carburising. In gas-carburising processes the carbon is supplied by the decomposition of a gaseous carbon compound which is either thermally unstable at the temperature of treatment, or is present in excess of its equilibrium concentration with respect to the products of its dissociation. Thus, in the case of methane, it is necessary to consider the equilibrium relationship for the reaction to ensure that at the temperature of treatment the methane concentration is in excess of the equilibrium value with respect to hydrogen, in order that carburisation may be promoted by the decomposition of the excess of the hydrocarbon. Hydrocarbons such as propane and butane, however are thermally unstable above approximately 500°–600° C., and tend to dissociate to the basic methane, setting free nascent carbon which is either absorbed by the steel or collects as sooty deposit. The end product, methane, is also a carburising gas, although its rate of decomposition is comparatively slow. Hydrocarbon gases are reducing and, provided that the rate of deposition of the carbon does not exceed that at which the carbon is absorbed by the steel, clean carburised work should be obtained. The degree of sooting is determined by the concentration of the hydrocarbon in the carburising atmosphere and this may be reduced by dilution with suitable media, such as air or a prepared burnt-fuel gas. Dilution of the hydrocarbon in this way reduces the tendency to sooting by promoting reactions between the decarburising gases, such as carbon dioxide, water vapour, hydrogen, and oxygen, and the hydrocarbon gas or the deposited soot. The concentrations of the decarburising gases thus influence the sooting tendencies of the carburising atmosphere, but precautions must be taken to ensure that decarburisation of the steel is not also obtained.

In the consideration of any gas carburising process it is necessary to bear in mind that carburisation is essentially a super-reaction process. Irrespective of the nature of the carburising gas, the atomic or nascent carbon is produced at the surface of the iron and not internally by the decomposition of the gas after diffusion. Researches into gas-metal reactions and diffusion processes have shown that a complex molecule is incapable of diffusion through a solid metal, except through large rifts or like defects in the structure. It may dissociate, however, into its individual atoms upon absorption on the metal surface, and atomic diffusion of the elements comprising the molecule may take place. Thus, in the case of carburisation by carbon monoxide, the primary reaction is the dissociation of the molecule of the gas at the surface into free carbon and oxygen atoms, the former being readily absorbed by the steel at the carburising temperature. Oxygen diffuses at a slower rate than carbon, so that most, if not all, of the atoms of this gas left free on the surface of the steel combine with another molecule of carbon monoxide or of any other reducing gas present. Some absorption of oxygen may also be obtained, especially in solid carburising, but in atmospheres free from oxygen-containing gases, the reaction at the surface of the steel is solely that of absorption and diffusion of the atomic carbon set free by the dissociating gas.

Experimental

The following three representative case-hardening steels were used for the experiments :

Low-carbon case-hardening steel (0·15% carbon).

3% nickel case-hardening steel (0·16% carbon, 3·17% nickel, 0·07% chromium).

Nickel-chromium case-hardening steel (0·13% carbon, 3·18% nickel, 1·10% chromium).

The majority of specimens carburised were in the form of round bars 150 mm. long, turned from 12·5–10 mm. dia. They were ground to a smooth and bright finish. Flat mild-steel plates, 40 × 12 × 3 mm. thick, ground to a smooth finish were used for a limited series of experiments with butane, and with a specially treated burnt town-gas atmosphere.

Carburisation of the flat plates was carried out in a butt-ended silica tube, $\frac{3}{4}$ in. in dia., heated in a thermostatically controlled vertical-tube furnace. For experiments on the round bars, use was made of a horizontal-tube furnace accommodating three silica tubes, each $\frac{3}{4}$ in. in dia. Thus, three tests could be carried out simultaneously at a constant temperature, under different conditions of gas flow or time of treatment, etc. The furnace was thermostatically controlled to within ± 3° C. of the required temperature, and in all tests the direction of flow of gas was reversed half-way through the carburisation period.

The various carburising and diluting atmospheres used in the experiments were :

Carburising Gases.

(*a*) Town gas.

(*b*) Butane.

(*c*) Propane.

(*d*) Partly burnt town gas, reheated over hot charcoal.

(*e*) Rich town-gas-air mixtures pretreated over hot charcoal.

(*f*) An atmosphere produced by the action of steam on hot charcoal.

Diluting Gases.

(*a*) Air.

(*b*) Partly burnt town gas.

(*c*) Partly burnt town gas, reheated over hot charcoal.

The temperature of carburisation selected was, in the majority of the tests, typical of industrial practice for the steel in question. Thus, whilst the carburisation of the mild steel in the partly burnt town-gas atmosphere, pretreated over hot charcoal, was studied at temperatures ranging from 750°–950° C., the work with hydrocarbon-containing atmospheres was carried out at 925° C. Both the alloy steels were carburised at 900° C. Among the factors studied were the composition of the carburising atmosphere, the rate of flow of the carburising gas, the time of treatment and the surface condition of the steel before carburisation.

The degree of carburisation was determined in a number of ways: (1) By measurement of the increase in weight of the steel per unit area of surface; (2) by chemical determination of the carbon content of the marginal layers of the carburised steel; (3) by metallographic estimation of the depth of the case; and (4) by depth-hardness measurements on the quench-hardened carburised steel.

TABLE II.—CARBURISATION OF 0·15% CARBON STEEL AT 925°C. FOR 5 HRS. IN TOWN GAS-BURNT TOWN GAS ATMOSPHERES.

Town Gas: 2·6% CO_2, 2·4% CnHm, 11·0% CO, 21·5% CH_4, 56·0% H_2, 2·0% H_2O, 4·5% N_2.
Burnt Town Gas: 4·7% CO_2, 9·1% CO, 14·7% H_2, 0·6% CH_4, 68·6% N_2, 2·3% H_2O.

Carburising Atmosphere		Weight Increase, mg./sq. mm. × 10^3	Case Depth, in.	Comments
Town Gas, %	Burnt Town Gas, %			
100	0	37·5	0·037	Heavy soot. Adherent. Non-uniform case.
75	25	31·5	0·032	Heavy soot. Adherent surface deposit. Non-uniform case.
50	50	27·2	0·030	Light soot. Readily brushed off. Uniform case.
25	75	19·2	0·017	Very light soot. Readily brushed off. Uniform case.
10	90	2·1	Trace	No soot.

Carburisation with Town Gas and Butane

In the carburisation of mild steel, using town gas as delivered from the mains, case depths of the order of 0·007 in./hr. for treatment up to 5 hrs. at 925° C. were obtained, but the results on the whole were inconsistent. Carburisation was always accompanied by heavy sooting of the steel, which resulted in non-uniformity in depth and carbon content of the case, and in some cases carburisation was prevented altogether. Dilution of town gas with partly burnt town gas decreases the amount of sooting, though only at the expense of a serious reduction in the rate of carburisation (see Table II). This is due to the fact that whilst town gas contains some very strong carburising constituents in the form of hydrocarbons, it is reduced to the status of a mildly-carburising gas by the presence of decarburising agents such as water vapour, carbon dioxide and hydrogen. Increasing additions of partly burnt town gas reduce, and eventually completely neutralise, the carburising power of the raw town gas. Removal of the decarburising gases from raw town gas results in a very strongly carburising atmosphere, as shown in Table III.

TABLE III.—CARBURISATION IN PURIFIED TOWN GAS.
Analysis: 9·0% CO, 52·5% H_2, 20·1% CH_4, 18·5% N_2.

Steel	5 hrs. at 925° C.		5 hrs. at 1,000° C.	
	Weight Increase, mg./sq. mm. × 10^3	Case Depth, in.	Weight Increase, mg./sq. mm. × 10^3	Case Depth, in.
Mild steel	56·0	0·060	86·0	0·080
Nickel steel	43·6	0·040	77·8	0·060
Nickel-chromium steel	—	—	80·1	0·076

Experiments with pure butane resulted in the rapid sooting of the reaction tube, so that even at very low gas velocities the passage of the hydrocarbon gas through the tube was stopped completely after a very short time. Further tests were carried out with butane diluted with air or with partly burnt town gas, the experiments being limited to the 3% nickel steel treated at 900° C. for a period of 5 hrs., and in a gas flow of 0·7 linear ft./min. In all tests soot was excessive.

Carburisation with Diluted Propane

Propane is available commercially as "Propagas," and may be obtained in cylinders or tanks in which it is liquified under pressure, 1 lb. of liquid propane giving approximately 8 cu. ft. of gas at N.T.P. The gas, having the formula C_3H_8 is a lower member of the methane series than is butane (C_4H_{10}), and has therefore less carbon available for sooting at elevated temperatures. The experiments with this gas have been associated with the elimination of sooting by dilution of the hydrocarbon with media such as air or a controlled atmosphere, whilst maintaining the maximum possible rate of carburisation.

TABLE IV.—CARBURISATION OF STEELS IN AIR-PROPANE ATMOSPHERES.
Rate of Gas Flow: 5 ft./min.

Propane, %	Time, hr.	0·15% C Steel at 925° C.			3·17% Ni, 0·16% C Steel at 900° C.			3·18% Ni, 1·1% Cr, 0·13% C Steel at 900° C.		
		Weight Increase, mg./sq. mm. × 10^3	Surface Carbon Content, %	Case Depth, in.	Weight Increase, mg./sq. mm. × 10^3	Surface Carbon Content, %	Case Depth, in.	Weight Increase, mg./sq. mm. × 10^3	Surface Carbon Content, %	Case Depth, in.
20	2½	10·0	0·38	0·007	4·5	0·16	—	10·0	0·57	0·025
20	5	13·1	0·41	0·015	5·5	0·18	—	12·6	0·58	0·033
20	10	21·3	0·50	0·024	7·0	0·21	—	19·5	0·64	0·043
24	2½	15·0	0·65	0·020	10·2	0·35	0·010	15·0	0·80	0·028
24	5	25·6	0·90	0·030	14·6	0·58	0·018	17·5	0·86	0·038
24	10	38·0	1·06	0·040	23·2	0·88	0·033	23·7	0·70	0·050
28	2½	23·0	0·85	0·027	16·2	0·65	0·020	19·5	0·69	0·034
28	5	38·5	0·94	0·038	21·5	0·76	0·030	22·3	0·74	0·042
28	10	61·7	1·12	0·058	41·2	1·05	0·038	29·2	0·84	0·060
32	2½	32·8	0·96	0·032	22·1	0·87	0·024	24·5	0·70	0·038
32	5	50·5	1·10	0·055	30·4	0·88	0·033	27·1	0·76	0·047
32	10	69·8	1·21	0·079	45·1	1·01	0·042	35·5	0·85	0·064

TABLE V.—CARBURISATION OF STEELS IN BURNT TOWN GAS-PROPANE ATMOSPHERES.

Burnt Fuel Gas: 4·8% CO_2, 10·8% CO, 15·6% H_2, 2·3% H_2O, 66·6% N_2.

Rate of Gas Flow: 5 ft./min.

Propane,	Time, hr.	0·15% C Steel at 925° C.			3·17% Ni, 0·16% C Steel at 900° C.			3·18% Ni, 1·1% Cr, 0·13% C Steel at 900° C.		
		Weight Increase, mg./sq. mm. × 10^3	Surface Carbon Content, %	Case Depth, in.	Weight Increase, mg./sq. mm. × 10^3	Surface Carbon Content, %	Case Depth, in.	Weight Increase, mg./sq. mm. × 10^3	Surface Carbon Content, %	Case Depth, in.
20	2½	29·5	0·92	0·027	13·2	0·37	0·010	10·0	0·52	0·027
20	5	40·7	1·11	0·037	16·5	0·47	0·017	13·5	0·54	0·037
20	10	64·5	1·22	0·055	23·0	0·59	0·027	17·5	0·59	0·050
24	2½	30·2	1·08	0·035	16·2	0·58	0·017	11·4	0·52	0·033
24	5	43·7	1·09	0·050	23·1	0·68	0·028	15·1	0·59	0·045
24	10	72·0	1·19	0·068	31·7	0·77	0·034	21·3	0·66	0·055
28	2½	31·5	0·98	0·038	18·6	0·64	0·023	12·5	0·56	0·036
28	5	46·6	1·17	0·057	26·9	0·78	0·032	16·5	0·63	0·048
28	10	73·3	1·22	0·078	32·0	0·83	0·040	22·4	0·71	0·058
32	2½	31·9	1·10	0·040	19·2	0·66	0·026	—	—	—
32	5	50·5	1·20	0·062	27·3	0·79	0·035	—	—	—
32	10	74·7	1·23	0·083	32·5	0·85	0·046	—	—	—

Carburisation of the plain carbon steel and the two alloy steels, was carried out at the appropriate temperatures in atmospheres containing 16–32% of the hydrocarbon, and for periods up to 10 hrs. The results are summarised in Table IV. Under any given conditions the maximum case depth was obtained with the nickel chromium alloy steel, whilst the nickel steel was found to give the lowest penetration of the three steels studied.

The explosive limits for air-propane mixtures are narrow, and lie within the range 2–9·5% of propane, but mixtures which give the maximum rate of carburisation are well outside this range. Dilution with air is inexpensive, and reduces the soot-forming tendency of the pure gas by combustion of a major portion of the propane added, thus, in effect, producing a complex atmosphere of the oxides of carbon, water vapour, nitrogen, hydrogen, and excess hydrocarbon.

Dilution of propane, either by a furnace flue gas or by a controlled atmosphere specially prepared in a generator by the controlled combustion of a fuel gas, also results in a reduction in the sooting accompanying carburisation. In effect, a carburising mixture is produced which is similar to that obtained by dilution of propane with air, but in this case it has the advantage of being under stricter control.

A series of experiments similar to those with air-propane mixtures were carried out with propane diluted with partly-burnt town gas, the composition of which is given, together with a summary of the results, in Table V. As in the case of dilution with air, a concentration of propane in excess of 30–32% did not materially improve the rate of carburisation, and above about 25% of propane sooting was excessive. But, on the whole, under comparable conditions of cementation, the sooting was less than in air-propane mixtures. Sooting was never completely eliminated, however, when cases of high carbon concentration were required, and in general the reduction in the amount of sooting with increasing dilution was not so marked as that obtained by similar dilution with air. The decarburising gases present in the burnt town gas tend to offset the carburising and sooting properties of propane, and by control over the amount of hydrocarbon added the type of case can be varied to suit requirements.

From the results for mild steel, it appears that burnt town gas-propane mixtures are more effective carburisers than air-propane mixtures of similar hydrocarbon content. However, in the case of the alloy steels, this is not so. It is probable that this difference is due to the fact that when the alloy steels were treated in the burnt-town gas-propane atmosphere, the soot produced was

TABLE VI.—EFFECT OF RATE OF FLOW OF ATMOSPHERE ON THE CARBURISATION OF STEEL.

Steel: 3·17% Ni, 0·16% C. Temperature: 900° C. Time: 5 hrs.
Diluting Gas: 4·8% CO_2, 10·8% CO, 15·5% H_2, 2·3% H_2O, 66·6% N_2.

Gas Flow, Ft./min.	Carburising Atmosphere.	Weight Increase, mg./sq. mm. × 10^3	Surface Carbon Content, %	Case Depth, in.
1	20% Propane—80% ...	11·8	0·36	0·010
2	Burnt Town Gas	14·7	0·40	0·013
3		16·6	0·46	0·018
5		16·5	0·47	0·017
1	28% Propane—72% Burnt	19·0	0·57	0·022
2	Town Gas	23·0	0·70	0·028
3		25·6	0·75	0·030
5		26·9	0·78	0·032

TABLE VII.—CARBURISATION OF STEELS IN CHARCOAL-TREATED BURNT TOWN GAS AND PROPANE.

Analysis of Diluting Gas: 20·8% CO, 1·2% H_2, 78% N_2. Rate of Flow: 5 ft./min.

Propane, %	Time, hr.	0·15% C Steel at 925° C.			3·17% Ni, 0·16% C Steel at 900° C.		
		Weight Increase, mg./sq. mm. × 10^3	Surface Carbon Content, %	Case Depth, in.	Weight Increase, mg./sq. mm. × 10^3	Surface Carbon Content, %	Case Depth, in.
2	2½	33·8	1·09	0·029	28·9	0·82	0·027
2	5	42·8	1·20	0·044	40·2	1·00	0·038
2	10	51·2	1·28	0·068	48·0	1·11	0·047
3	2½	—	—	—	30·6	0·95	0·030
3	5	—	—	—	43·7	1·08	0·042
3	10	—	—	—	50·1	1·16	0·051
4	2½	44·4	1·19	0·034	33·2	1·04	0·032
4	5	54·2	1·25	0·054	44·1	1·13	0·042
4	10	66·4	1·34	0·076	51·0	1·16	0·054
6	2½	46·3	1·19	0·037	—	—	—
6	5	58·1	1·24	0·066	—	—	—
6	10	71·0	1·30	0·083	—	—	—

TABLE VIII.—CARBURISATION OF 0·15% CARBON STEEL IN VARIOUS ATMOSPHERES.
Carburisation: 5 hrs. at 925° C. Rate of Gas Flow: 5 linear ft./min.

Carburising Atmosphere.	Composition of Carburising Atmosphere.									Weight Increase mg./sq. mm × 10^3	Surface Carbon Content, %	Case Depth, in.	Appearance of Carburised Work.
	CO_2, %	C_nH_m, %	CO, %	H_2, %	CH_4, %	H_2O, %	N_2, %	O_2, %	C_3H_8, %				
Raw town gas	2·6	2·4	11·0	56·0	21·5	2·0	4·5	—	—	37·5	0·80	0·037	Heavily sooted.
Purified town gas	—	—	9·0	52·5	20·1	—	18·5	—	—	56·0	1·20	0·060	Lightly sooted.
32% Propane, 68% air	—	—	—	—	—	—	53·5	14·5	32·0	50·5	1·10	0·055	Heavily sooted.
32% Propane, 68% burnt town gas	3·5	—	7·4	10·6	—	1·6	45·0	—	32·0	50·5	1·20	0·062	Heavily sooted.
6% Propane, 94% purified burnt town gas	—	—	19·6	1·1	—	—	73·3	—	6·0	58·1	1·24	0·066	Clean, matt.
Chemically-purified burnt town gas	—	—	19·7	15·0	—	—	74·3	—	—	22·0	0·88	0·032	Clean, bright.
Charcoal-treated burnt town gas	—	—	21·3	1·2	—	—	77·5	—	—	47·0	1·13	0·035	Clean, matt.
Reacted town gas	—	—	24·4	35·6	1·0	0·1	39·0	—	—	45·0	1·00	0·040	Clean, matt.
98% Reacted gas, 2% Propane	—	—	23·8	34·8	1·0	0·1	38·3	—	2·0	50·2	1·16	0·058	Clean, matt.
Steam-charcoal gas	—	—	49·0	48·5	2·5	—	—	—	—	48·8	1·03	0·050	Clean, matt.

in the form of a dense shell which could in some instances be removed as a tube from the cylindrical specimens. This hard carbon deposit formed a compact case around the steel and reduced the rate of carbon absorption. The difference in the nature of the soot deposits was probably associated with the catalytic activity of the different steels in promoting the rapid pyrolysis of the hydrocarbon.

The results on the effect of the linear rate of flow of the carburising gas upon the degree of carburisation obtained are given in Table VI. Below 2·0 linear ft./min. there was a slight decrease in the amounts of soot and oil dissociation products formed. Above this rate of flow, there was very little increase in the rate of carburisation.

Experiments were carried out to determine the maximum concentration of propane which could be tolerated at any temperature before sooting was obtained, the diluting gas being burnt town gas processed over hot charcoal. Specimens of mild steel measuring approximately $20 \times \frac{3}{4} \times \frac{1}{8}$ in. were heated in a silica tube in which the steel was so located that a temperature gradient existed from the middle of the bar to each end. The carburising gas was used only when the temperature conditions were stabilised, pure hydrogen being employed during both the heating and the cooling of the steel. In all cases the time of heating in the carburising gas was limited to 2 hrs. The results are given in Table VII.

The experiments indicated that the composition of the charcoal treated gas, and in particular its hydrogen content, has little bearing on the minimum temperature at which carburisation is obtained without sooting. The direction of the gas flow relative to the steel appears to be of some importance. Direct impingement of the carburising gas on the steel surface immediately on entering the reaction chamber promotes sooting at temperatures at which no sooting is obtained when the gas mixture does not reach the steel surface immediately on entry. Thus, in commercial installations, the use of forced circulation inside the carburising container would promote the heating of the freshly charged gas before it meets the hot steel surface, and would tend towards a reduction in the amount of soot deposited on the work.

All the mild-steel specimens treated in atmospheres containing 4% or less of propane were free from sooting, and were bright after treatment, although slightly etched and rather duller than in the as-received condition. Both the nickel and the nickel-chromium steels, under the conditions of test, were slightly sooted after carburisation in atmospheres containing more than 2% and $1\frac{1}{2}$% of propane, respectively. Where sooting did take place it took the form, in the majority of tests, of merely a loose surface film which could be removed readily by brushing, leaving a clean, matt underlying surface. With forced circulation instead of streamlined flow, which was the condition obtaining in all the tests, it is very probable that higher concentrations of propane could be tolerated at the temperatures used in the experiments without sooting resulting. Carburisation of mild steel in various atmospheres is given in Table VIII.

The Nature of the Steel Surface and its Effect upon the Rate of Carburisation

The rate of carburisation in any atmosphere can be influenced considerably by the condition of the steel surface, and this aspect of the subject has been investigated, using a carburising gas of burnt town gas from which the carbon dioxide and water vapour had been completely removed. Flat mild-steel specimens were used for the experiments, carburisation being carried

TABLE IX.—EFFECT OF SURFACE CONDITION OF 0·15% CARBON STEEL ON THE DEGREE OF CARBURISATION IN DRY CO_2—FREE BURNT TOWN GAS.
Carburising Temperature: 950° C. Carburising Time: 5 hrs.

Gas Flow, ft./min.	Surface of Specimen.	Carburising Atmosphere.	Weight Increase, mg./sq. mm. × 10^3
1	Abraded with No. O emery cloth	10·7% CO, 15% H_2, 74·3% N_2	13·9
1	Sand-blasted	" " "	12·0
1	Shot-blasted	" " "	12·1
1	Pre-oxidised for 10 min. at 950° C.	" " "	15·3
1	Oxidised for 1 min. at 1 hr. intervals during carburising	" " "	8·45
1	Pre-oxidised; oxidised again for ½ min. after 2½ hours carburisation	" " "	13·10
5	Abraded with No. O emery cloth	" " "	21·60
5	" " "	0·05% O_2 } 10·7% CO, 15% H_2	18·20
5	" " "	0·10% O_2 } remainder N_2	13·70

out in all cases at 950° C. for 5 hr. Results typical of those obtained are given in Table IX. It is shown that roughening the surface by heating the steel in an atmosphere in which inter-gas reactions are promoted by the steel, markedly increases the activity of the surface, and speeds up the rate of carburisation.

The temperature of carburisation and its effect on the rate of carburisation is also discussed, and it is shown that at and just below the upper critical point of the steel being treated, the rate of absorption of carbon falls off markedly. It is suggested that this is associated with grain refinement during heating up to the Ac_2 part, which in turn promotes a sharp gradient between the case and the core.

The relative activities of solid carbon and of cementite are discussed, and it is concluded, from observations made in the course of the investigations, that below about 800° C. solid amorphous carbon is stable with respect to cementite, but that above this temperature the reverse is the case.

A Standard Bush Product

Bi-metal strip comprising leaded bronze and steel, provides the bearing qualities and strength necessary.

THE great practical significance of standardisation is generally appreciated and it has been effected to a considerable degree in light engineering and electrical components, but the advantages to manufacturer and user resulting from the incorporation of standard components in an engineering product are such that the policy is worthy of further development. Among the many components which are now standardised bushes are not the least important, and in this field a bi-metal bush has been developed and standardised which has many interesting and useful features.

The bi-metal strip pressed and rolled to form a bush.

This standard bush product, by The Glacier Metal Co. Ltd., makers of plain bearings and of bronze and white metal alloys, is the result of long research and experiment in engineering and metallurgical fields, conducted with the following objects in view :

1. To develop a standard bush which would be a great improvement on phosphor-bronze bushes and better suited for a wide number of applications.
2. To plan a manufacturing process which would make possible very rapid production of such bushes at low cost.
3. To make them in a sufficiently wide range of standard dimensions to meet the general requirements of all branches of the engineering industry, and
4. To provide the full range ex stock.

The investigation resulted in the development of bi-metal material comprising steel and lead-bronze strip. Lead-bronze is recognised as a bearing bronze with considerably better anti-friction properties than phosphor-bronze. On the other hand, lead-bronze in cast bush form has lower mechanical strength than phosphor-bronze. The combination of the bearing alloy and steel backing provides a bush with appreciably improved bearing qualities, permitting the use of a softer shaft, and giving the mechanical strength desired.

The lead-bronze lining is firmly bonded to the steel backing by means of a sintering process, and the steel part of the strip is copper-plated so that rusting is avoided. A series of pressing operations convert the bi-metal strip into butt-jointed bushes through a final rolling operation. The making of bushes from rolled bronze is not new, such bushes have been used by the motor industry for many years, but, generally, only for light-load applications. The use of the bi-metal strip in the Glacier wrapped bushes greatly increases the field of application since they are suited to heavy as well as light loads.

These bushes are stocked in nearly 200 standard dimensions between $\frac{3}{8}$ in.–$2\frac{1}{4}$ in. bore, and $1\frac{5}{32}$ in.–$2\frac{7}{16}$ in. diameter, with four or more standard lengths to each bore size. A similar range in metric sizes is being manufactured, particularly for the export market. They are produced to diameters which ensure the correct interference fit when they are pressed into housings machined to Newall " B " limits for holes. The bush is inserted in the housing by means of an arbor with a pilot. The bush is assembled on the pilot, carefully inserted into the housing, and the arbor is pressed home by means of a press, a carefully used vice, or by the use of draw-bolts. As supplied, the bushes will be found on assembly to have a finishing allowance in the bores.

From both the commercial and technical points of view these bushes would appear to offer advantages over cast bushes. Briefly it can be stated that they are less expensive due to large-scale production by a rapid process. They are lighter and make possible the use of a smaller housing. They combine the strength of steel with the bearing qualities of lead-bronze. The lead content of the bearing alloy lowers friction, reducing running temperatures and, in order further to minimise the possibility of seizure, a slight degree of residual porosity is deliberately left in the lead-bronze lining, so that oil is retained in the pores during running.

Correspondence

Open Rotary Furnace

The Editor, METALLURGIA.

Sir,

I have followed with interest the discussion on the application of the open rotary furnace referred to by Mr. C. F. Jinks, in your April issue, and the modified form described by Mr. H. Landseer-Bailey in your June issue. It is probable that further information on the subject will be of interest to your readers.

About seven years ago I was approached, in the capacity as a consultant on metallurgical matters, to investigate and effect a remedy for the high losses in melting aluminium swarf by the use of a converted 2 to 3 cwt. concrete mixer. As arranged at that time the flame from the burner was directed into the mass of unprotected swarf, with the obvious result that the melting shop was soon filled with fumes. Later I inspected, in the same capacity, another plant, which was equipped with three or four of these small converted concrete mixers, which were used for the recovery of aluminium from dross. The dust and fumes emanating from one of these furnaces made the shop very uncomfortable for everyone working in it. Still later, I learned that several of these small furnaces were working under somewhat similar conditions in the Coventry area.

The remedy for these losses of valuable metal is the application of a melting method similar to that described by Mr. Jinks.

The simplicity of this furnace, its labour saving and its high yield, fascinated me deeply, and I studied its possibilities intensively. As a result of this I approached Messrs. Winget, as well as the Ministry of Supply, with a view to the development and exploitation of this type of furnace in the national interest.

In co-operation with the late sales manager of Winget's, improvements were considered but the collaboration was interrupted by his sudden death before they could be developed. The intention was to broaden the basis of application of this furnace and to provide practical information, which would enable operators to obtain good results in the recovery of aluminium from residues and in the general working of the furnace. For this purpose I prepared an extensive paper on the subject, which was not published on account of the interruption.

The possibilities of this furnace, which can be regarded as the furnace for the recovery of usable metal from aluminium residues, are not yet fully exploited, but are discussed in detail in the paper referred to. A useful modification of this furnace is the application of Mr. Landseer-Bailey's idea. The furnace can handle a charge of one ton, and the fumes emanating from the use of salts can be carried away by a hood covering every position of the furnace.

Mr. Jinks reported to having obtained the satisfactory result of one cwt. of metal per hour by low melting costs. It is noteworthy that at another recovery plant in continuous melting five cwts. of metal per man hour could be recovered. This figure had been determined in theory and was obtained in practice. The material charged comprised fine aluminium turnings; they were centrifuged, but still contained 3 to 5% moisture, and the yield of recovered metal varied between 91 to 94%. It is very doubtful whether such results can be obtained by any other means, results which are mainly due to rapid melting.

Although the small furnace of two to three cwt. capacity had been in use for many years, operating in a very crude way, without the intensive consideration of its possibilities for treating residues, this furnace may never have come out of its obscurity because of the crude and prohibitive method of using it for the recovery of usable metal from aluminium dross.

Yours faithfully,

H. O. LUEDTKE,
Metallurgical Engineer,

Holifast Road,
Sutton Coldfield,
Warwickshire.
9th August, 1946.

Centrifugal Oil Purifiers

THE use of the centrifugal purifier for treating cutting oils and grinding coolants in the modern workshop is now recognised as a most important aid to efficiency, on account of its threefold advantages: the elimination of dermatitis, improved machining, and direct saving in costs.

Oil dermatitis is mainly caused by small punctures of the skin being contaminated by impure oil, and such punctures are very largely due to the fine metallic particles in the cutting oil. When oil is not regularly purified, the small metal chips formed during the cutting operation are inevitably rendered still finer by recirculation past the cutting edge, and skin punctures from this source become almost unavoidable. The regular cleaning of bulk oil supplies, and purification of the oil recovered from swarf, combined with sterilisation, practically obviates the danger of oil dermatitis, and consequently increases the confidence and contentment of employees.

The removal of fine metal particles, by regular purification of the cutting oil, enables higher cutting speeds to be used, considerably increases the life of tools between grinds, and provides a finer surface finish. The importance of this can readily be appreciated when valuable tools, such as those used in hobbing and gear cutting, are used, and also in cases where tolerances are close. Clean cutting oil is an essential for the successful operation of automatic and semi-automatic machines on which tool wear is an important factor, and the purification of cutting oils is also necessary to remove dust, grit and water which are collected by the oil in use, in addition to minute metal particles, and which inevitably penetrate into the bearing surfaces of a machine.

Centrifugal purifiers ensure that a very high percentage of the oil entrained in swarf is recovered, purified and sterilised for re-use, an advantage which is of particular importance in the case of the relatively costly straight cutting oils. Various methods are generally employed for separating oil from the chips, including centrifuging in a basket-type extractor, spraying with hot water, or steaming, but these treatments do not dispose of the impurities contained in the reclaimed oil, and consisting chiefly of fine metallic particles and acid sludge formed by breakdown of the oil in use. Complete and efficient purification can only be secured by means of a centrifugal purifier. A range of machines made by Hopkinsons Limited has proved successful in connection with these duties, and in a typical layout containing one of these purifiers, the drainage from the chip dump and the oil recovered in

the chip extractor are led to a dirty-oil tank, from which the bulk of the heavy dirt can eventually be drained. From this tank, the oil is led to a heater where its temperature is raised to 180°–200° F.; this not only lowers the viscosity of the oil, but also sterilises it. The oil now passes to the centrifugal purifier, where all dirt, water and sludge are removed.

The "three-purpose" bowl of the machine is carried on a vertical shaft running in ball bearings, the drive being through gears, lubricated by oil mist. All parts of the bowl are protected from corrosion, and the speed of the bowl, varying from about 10,000 r.p.m. in the case of the smallest machines to about 5,000 r.p.m. in the case of the largest units, is checked by a speed counter.

For the grinding compounds, used on all modern precision grinding machines to carry away the heat generated, and to flush metallic particles and loose abrasive from the line of contact between the stock and grinding wheel, a settling tank is incorporated in the machine base to allow settlement of the heavier particles. Owing to the high circulation rate, the limited quantity of coolant, and the fact that much of the material removed is in a very finely divided state, this is not very effective. These harmful impurities are, however, cheaply and effectively removed by a Hopkinson centrifugal clarifier, which thereby permits the use of softer wheels and materially increases the life of the wheels, produces a better finish on the work, and cuts production times. In addition, wear on coolant pumps is reduced, and the useful life of the coolant prolonged almost indefinitely.

The Institute of Metals

THE autumn meeting of the above Institute will be held in London on September 10 and 11, 1946. The business meeting and technical sessions will be held at the Institution of Civil Engineers, Great George Street, London, S.W. 1, commencing at 2-30 p.m., on September 10. Two technical sessions will be held simultaneously the following morning, in the Lecture Theatre and the South Reading Room of the Institution of Civil Engineers. The papers to be presented immediately after the business meeting are as follows :—

D. McLean and L. Northcott : "Antimonial 70 : 30 Brass."

D. C. G. Lees : "The Hot-Tearing Tendencies of Aluminium Casting Alloys."

H. W. L. Phillips : "The Application of Some Thermodynamical Principles to the Liquidus Surface of Alloys of Aluminium with Magnesium, Silicon and Iron."

On September 11, commencing at 10 a.m., in the Lecture Theatre, the following papers will be presented and discussed :—

Marie L. V. Gayler : "The Ageing of a High-Purity Aluminium Alloy Containing 4 % of Copper."

Marie L. V. Gayler : "The Cold-Working of a High-Purity Aluminium Alloy Containing 4 % of Copper and its Relation to Age-Hardening."

L. E. Benson : "Control of Internal Stresses in Heat-Treated Aluminium Alloy Parts."

R. J. M. Payne : "Inverse Segregation in Cast Magnesium Alloys."

G. J. Metcalfe : "Intercrystalline Corrosion of Aluminium-Magnesium Alloy Rivets."

In the South Reading Room, commencing at 10 a.m., the following papers will be presented and discussed :—

J. W. Cuthbertson : "The Resistance to Corrosion by Sea-Water of Some Alpha-Tin and Alpha-Tin-Aluminium Bronzes."

A. Latin : "Capillary Flow in the Soldering Process and Some Measurements of the Penetration Coefficients of Soft Solders."

M. Cook and W. O. Alexander : "The Physical Properties and Temper-Hardening Characteristics of Copper-Nickel-Manganese Alloys."

H. J. Gough and D. G. Sopwith : "Inert Atmospheres as Fatigue Environment."

High Frequency Generator

A 25 kw. high frequency heating generator suitable for eddy-current heating of metals or for dielectric heating of non-conductors has been introduced by the G.E.C. It is eminently suitable for many heating processes and though the variation of individual cases is so large that it is difficult to give definite performance figures, the following examples are a general guide to what this set can do under average conditions :—

Heat 40 lb. of ferrous or 18 lb. of non-ferrous metal through 100° C. per minute.

Harden 10 sq. ins. of steel surface to a depth of 30 thousandths of an inch, taking 5–10 secs.

Harden 2–3 sq. ins. of steel surface to a depth of about 10 thousandths of an inch taking about one second.

Preheat 12 lb. of plastic material in one minute.

Dry off 50 lb. of water from a charge per hour.

Heat 12–15 lb. of seasoned wood 100° C. per minute.

Strongly built and designed for constant use under conditions likely to be met in factory or workshop, the generator is constructed so that only the front face and right-hand side need to be free. In consequence, it can be placed conveniently in a corner or backed against a wall. The generator is completely self-contained, requiring connection only to the supply mains and to the heating coils or plates. The output of 25 kw. is a continuous rating with substantially resistive loads. The operating frequency range is 2–15 Mc/s, the generators being sent out adjusted to the most suitable frequency for each class of application.

Reparations from Germany

THE Inter-Allied Reparations Agency, Brussels, has just completed the first allotment between the Western Powers of capital plant which is to come as reparations from Germany. In this allotment, Britain has secured equipment from the works of Fritz Mueller, Waldrich, Heniel and Lueg, Jurbelwellenwerk and Hanseatische Kettenwerke, mainly very urgently needed machine-tools.

The procedure for disposing of German reparations plant, of which this is the first minor instalment, will normally follow the lines of the existing disposal schemes for the sale of Government surplus machinery, and the machines will be sold and delivered to buyer's works, and will be expected to realise prices roughly equivalent to those ruling in the country for corresponding equipment in the same second-hand condition.

"COPPER DATA," "Bearing Bronzes," "German Copper and Brass Welding Practice," "The Resistance of Copper to Soil Corrosion" and "Classification of Copper and Copper Alloys" have been reprinted and will be supplied free of charge by the Copper Development Association, Kendals Hall, Radlett, Herts.

Some Recent Appointments and Staff Changes

MR. M. W. THRING has been appointed Head of the Physics' Department of the British Iron and Steel Research Association. Mr. Thring has been with the British Coal Utilisation Research Association since the days when it grew out of the Research Department of the Combustion Appliance Makers' Association. For the last few years he has been in charge of the Furnace Research Section and Combustion Research Laboratories, and for the last eighteen months he and a large part of his team have been engaged on a co-operative research with Dr. J. H. Chesters and the United Steel Companies Ltd., into the study of flames in furnaces.

DR. W. C. NEWELL, until recently on the staff of the Brown-Firth Research Laboratories, Sheffield, has been appointed Head of the Steel Castings Division of the British Iron and Steel Research Association. Dr. Newell received his earlier training and research experience at the Royal College of Science.

MR. E. L. DIAMOND has been appointed Mechanical Engineer to the Plant Engineering Division of the British Iron and Steel Research Association. Mr. Diamond graduated in engineering with honours at Kings College, London, in 1922, and then became a pupil of the late Sir Henry Fowler at Derby locomotive works of the Midland Railway, later assisting in experimental work. Since 1926 he has been on the technical staff of the Institution of Mechanical Engineers, except during the war years, 1940-1945, when he became Inspector (Components) in the Inspectorate of Fighting Vehicles. He has published a number of papers on technical locomotive problems, and received the degree of M.Sc.(Eng.) in 1936, for some of this work.

MR. G. A. WHIPPLE, M.A., M.I.E.E., F.Inst.P., has been appointed Managing Director of Adam Hilger, Ltd. in succession to Mr. Twyman. Mr. Whipple, who is also Managing Director of E. R. Watts and Son, Ltd., is the son of Mr. Robert S. Whipple, Chairman of the Cambridge Instrument Company. After graduating at Cambridge, he carried out research work in Germany and in this country. He has served on the Council of the Institution of Electrical Engineers, and has been Hon. Secretary of the Scientific Instrument Manufacturers' Association for the last six years. He is a member of the Boards of Governors of Northampton Polytechnic and the National College of Horology.

MR. A. W. BURRELL, who has for several years represented the Cambridge Instrument Co., Ltd. in the Home Counties, has been appointed Resident Engineer for that company in the Manchester district. In that capacity he succeeds Mr. J. L. Orchard, who has retired from active work on reaching the age limit.

MR. A. L. MORRISON, a Director of Kayser, Ellison and Co., Ltd., has been appointed Representative for that firm in the Midlands and Scotland in succession to Mr. Herbert Francis, M.B.E., who has retired after nearly 67 years' service with the company. Mr. Morrison had the charge of sales in London and the South of England. Mr. R. E. Linton, who for several years assisted Mr. Morrison in London, is now in charge of the London office.

MR. FRANK TWYMAN, F.Inst.P., F.R.S., has been appointed technical adviser to Adam Hilger, Ltd., and to their associates, E. R. Watts and Son, Ltd. Mr. Twyman joined Hilger's in 1898; became Manager of the firm on the death of Mr. Otto Hilger, in 1902, and Managing Director of the company on its incorporation in 1904, a position which he has resigned. He, however, remains Chairman of Hilger's. Under Mr. Twyman's direction this firm has specialised in the manufacture of optical instruments for research in physics and chemistry, and for the control of industrial processes. These instruments have contributed in no small measure to knowledge in atomic and molecular physics, and have brought optical glass work to a high level of perfection. In recognition of the value of these instruments, many of which were designed by Mr. Twyman, he was in 1924 elected a Fellow of the Royal Society.

MR. H. J. STARLING, a London representative of the Cambridge Instrument Co., Ltd., has been appointed Resident Engineer for that company in the newly-formed Nottinghamshire district, which covers Nottinghamshire, Cambridgeshire, Lincolnshire, Leicestershire, Northamptonshire, Rutland, Huntingdonshire, Norfolk and Suffolk.

DR. C. L. WILSON has been appointed to a Lectureship in Chemistry with special reference to Micro-Chemistry, in the Queen's University of Belfast. Dr. Wilson is a frequent contributor to the microanalysis features in this journal.

MR. S. PERCIVAL has been appointed by Zinc Alloy Rust Proofing Co., Ltd., as Manager of their works in Shakespeare Street, Wolverhampton. He succeeds Mr. J. C. Mills, and will continue an already long record of service with the company.

MR. R. A. HACKING, M.Sc., F.I.M., has been appointed by Richard Thomas and Baldwins, Ltd., to deal with and advise on all that company's iron and steel production, including new construction and development. He has resigned his position as a special Director of Dorman, Long and Co., Ltd., to take up this appointment.

MR. D. N. LOWE has succeeded Dr. O. J. R. Howarth as Secretary of the British Association for the Advancement of Science, who has retired after serving in that capacity since 1909. Mr. Lowe served as Assistant Secretary before being seconded during the war to work for the Government.

MR. T. MAKEMSON has been released from his position as Director of Iron Castings in the Iron and Steel Control, and has returned to his post as Secretary of the Institute of British Foundrymen. He will continue to act as honorary adviser on iron castings to the Ministry of Supply.

MR. R. GRESHAM COOKE, M.A., Secretary of United Steel Companies, Ltd., since 1939, has been appointed Director of the Society of Motor Manufacturers and Traders, Ltd. The new Secretary of United Steel Companies, Ltd. will be Mr. R. Peddie, B.A., who has been with the firm for the past three years.

SIR JOHN FISHER and Mr. J. K. Vaughan-Morgan have been appointed Directors of the Barrow Hematite Steel Company, Ltd.

Some Recent Appointments and Staff Changes—*Contd.*

DR. EDWIN GREGORY, Chief Metallurgist for Edgar Allen and Co., Ltd., has been elected President of the Institution of Engineering Inspection in succession to Major-General C. A. Woods, C.B., C.B.E., M.C.

SIR JOHN LENNARD-JONES, K.B.E., Ph.D., D.Sc., F.R.S., has relinquished his appointemnt as Director-General of Scientific Research (Defence) in the Ministry of Supply with effect from July 31, 1946. Sir John, who has been on loan to the Ministry since the outbreak of war, will return to his post at Cambridge University. The University has agreed to make his services available on a part-time basis as Chief Scientific Adviser to the Ministry of Supply.

MR. NORMAN ROWBOTHAM, C.B.E., F.R.Ae.S., W.r.Ex., M.I.A.E., M.I.P.E., a Director of Bristol Aeroplane Co., Ltd., has been elected President of the Institution of Production Engineers.

MR. W. S. SPICER, Chief Buyer and Stores' Superintendent of Hadfields, Ltd., has been appointed a Local Director of the company.

MR. TIM SIMS, the well-known Canadian pilot and aero-engineer, has been selected to fly the Bristol "Freighter," which is to tour Canada and the Americas in the course of the longest of the series of demonstration flights planned to reveal the performance and potentialities of the Bristol Aeroplane Company's multi-purpose aircraft. Mr. Sims, who is Vice-President of Bristol Aeroplane Engines, Ltd., Montreal, Canadian agents of Bristol Aeroplane Company, has been flying aircraft of all types for nearly 20 years, and has extensive knowledge of the airways of North and South America.

SIR ALEXANDER DUNBAR has relinquished his Directorships of the English Steel Corporation, of which he has been Chairman since November last, Vickers-Armstrongs, Ltd., and of Firth-Vickers Stainless Steels, Ltd., of which he was also Chairman. Resulting changes in the directorate of the Vickers group are announced as follows:

Lt.-General Sir Ronald Weeks, K.C.B., has been appointed Deputy-chairman of Vickers, Ltd., and Chairman of English Steel Corporation.

Sir Frederick Yapp has relinquished his office of Managing-director of Vickers, Ltd., but retains his seat on the board. He has retired from the boards of Vickers-Armstrongs, of which he was Chairman, and the English Steel Corporation.

Commander Sir Robert Micklam, C.B.E., has been appointed Chairman of Vickers-Armstrongs, retaining the office of Managing-director of engineering works and shipyards.

Major H. R. Kilner, M.C., has been appointed Deputy-chairman of Vickers-Armstrongs, retaining the office of Managing-director (aviation).

Mr. C. E. Holstrom has been appointed Managing Director of Firth-Vickers Stainless Steels, Ltd. with effect from September 30, when Mr. James Wortley Fawcett retires from that position and from the board.

Mr. J. T. Dewar, formerly General Manager of the Engineers' Tool Department, is appointed Director in charge of production, and Deputy Managing Director. Mr. J. V. Hall and Mr. R. Staton are appointed special Directors.

MR. W. R. VERDON SMITH, a Director of the Bristol Aeroplane Co., Ltd., Rotol, Ltd., and A. F. N., Ltd., and son of Sir W. G. Verdon Smith, Chairman of the Bristol Aeroplane Company, has been elected President of the Society of British Aircraft Constructors for 1946-47, in succession to Mr. Arthur Gouge. At 33, Mr. Verdon Smith is the youngest chairman ever to have been elected by that society.

Personal

DR. WILLIAM GRIFFITHS, Chairman and Managing Director of The Mond Nickel Company, Limited, was among those knighted by the King at an Investiture held on July 23. This honour, which was announced in the Birthday Honours, was conferred for public services rendered by Sir William, and in particular for his valuable work on metallurgical problems during the war.

Sir William and Lady Griffiths at the Investiture at Buckingham Palace.

Sir William's association with The Mond Nickel Company, Limited, began in 1926, and from 1928 to 1945, as Manager of the Research and Development Department, he was responsible for the development of many new uses for nickel. He became Chairman and Managing Director of the company in 1945, and in the same year was appointed a Vice-President in charge of operations in Great Britain and European activities, of The International Nickel Company of Canada Limited.

HUGH WARREN, of Thurlaston Grange, Rugby, Managing Director of the BTH Company, and formerly Director of the company's Research and Engineering, has received the Degree of Doctor of Science, Honoris Causa, at Birmingham University.

Dr. Warren is a member of several learned institutions and of scientific and industrial committees. He is the author of numerous technical books and papers. His Doctorate recognises in particular his work in industrial research and technical education. He is a Director of the Metropolitan-Vickers Electrical Company and of several other companies connected with Associated Electrical Industries Limited.

SIR WILLIAM GRIFFITHS, Chairman and Managing Director of Mond Nickel Co., Ltd., has been nominated as a representative of that organisation to the Council of the Copper Development Association.

Metallurgical Expansion in Soviet South

By I. Andronov.

ANOTHER large blast furnace has been put into operation in the south of the Soviet Union thus keeping pace with the intensive redevelopment of industry. This furnace, at the Dnieprodzerzhinsk Steel Works, was built in 1932 and for about ten years continuously smelted iron until the Dnieper area, captured by the Germans, was wrecked. Restoration involved practically reconstruction of the blast furnace. But before starting upon the work of erection nearly 10,000 cubic yards of rubble and 7,500 tons of ore and slag had to be removed. Some 2,500 cubic yards of bricks were laid and over 1,500 cubic yards of concrete and ferro-concrete work constructed. The builders dismantled and mounted anew some 3,500 metal structures and put down more than seven miles of tubing, two miles of railway tracks and six miles of cables.

Engineers introduced technical innovations and improvements developed in Soviet and world metallurgy in recent years with the result that the present blast furnace, in respect of technical equipment, is superior to its pre-war state.

With this furnace in operation the Dnieprodzerzhinsk works can double its output of pig-iron. This is an event in the post-war rehabilitation of the entire iron and steel industry in the Soviet south. As a result, it is believed that by 1950 the Dzerzhinsk works will surpass the pre-war level output of pig-iron by 62%, steel by 110% and rolled metal by 77%.

Rapid progress is being made in the restoration of plants in Soviet south situated in the steppes of the Ukraine and Black Sea area, between the rivers Don and Dnieper. Month by month, more workers, materials and equipment are being employed and more capital invested in Dnieprodzerzhinsk, Dniepropetrovsk, Zaporozhye, Krivoi Rog, Nikopol, Stalino, Makeyevka, Yenakiyevo, Kramatorsk, Voroshilovgrad, Mariupol, Tagan Rog and other southern cities of the Soviet Union.

One large blast furnace, seven open-hearth furnaces, six rolling mills, a power station, a concentration plant and numerous auxiliary shops of the Makeyevka Iron and Steel Works which were blown up by the Nazis, have already been rehabilitated and are working. The restoration of another of its blast furnaces has just been completed and the restoration of three open-hearths, a blooming mill and three rolling mills is reaching completion. By 1950 this enterprise is expected to increase its pre-war output of pig-iron by 20%, steel by 38% and rolled metal by 30%.

Among other important southern steel and iron mills undergoing rapid reconstruction are the Zaporozhstal works with its blast- and open-hearth furnaces, and slabbing and sheet mills, producing sheet steel for the rapidly developing automobile industry; the Stalino High Grade Steel Works; the Azovstal Works with its blast- and open-hearth furnaces and rolling mills; and numerous tube-roiling works. The southern coke and chemical industry is also rapidly being restored.

To restore the pre-war level of production of pig-iron, steel, rolled metal, ore and coke, scores of southern enterprises are being re-equipped including 17 large steel works and seven tube-rolling and pipe-casting plants. In the next five years, 30 blast and 76 open-hearth furnaces and 54 rolling mills will be erected and put into operation in these works. In 1950 the Southern steel and iron industry is expected to produce 9,700,000 tons of pig-iron and 8,800,000 tons of steel. The productive capacity of many of the works will be augmented by the reconstruction and modernisation of restored plants and the construction of new ones.

As a result of the progress so far achieved it is believed that the pre-war level of the production of steel and rolled metal will be attained within three years, and pig-iron within four years from the end of the war.

Conference on German Patents

A conference to consider the question of the future treatment of German-owned patents in Allied countries took place in London from July 15 to 27. Delegates attended from Australia, Belgium, Canada, Czechoslovakia, Denmark, France, Luxembourg, Netherlands, Norway, Union of South Africa, United Kingdom, United States of America. The chairman was Sir Harold Saunders, the Comptroller of Patents in the United Kingdom.

Patents taken out by Germans exist in varying numbers in all countries of the world. Complete unanimity prevails among the Allied Nations that in no circumstances shall any such patents within their territories revert to the former German owners, and the question as to how such rights shall in future be disposed of presents many difficulties. A strong sentiment prevails that it would be unfortunate if the continued existence of these patents should constitute an obstacle to international trade.

As a result of the discussion at the conference the representatives of France, the Netherlands, the United Kingdom and the United States of America have signed an accord which will have the effect of making all patents of former German ownership now controlled by their Governments, and in which there is no non-German ownership now controlled by their Governments, and in which there is no non-German interest existing on August 1, 1946, available within their respective territories to all nationals of the countries party to the accord without payment of royalties or without any requirement to manufacture within the country where the patents exist. The representatives of Australia, Canada, Czechoslovakia, and the Union of South Africa have agreed to recommend to their respective Governments that the accord should also be signed on behalf of those Governments.

Representatives of Belgium, Denmark, Norway and and Luxembourg feel that the special difficulties which exist in their countries render it necessary for their Governments to give a more detailed and closer examination of the provisions of the accord before coming to a decision as to their policy.

As stated above, the accord has been signed by four countries, and it remains open for signature by other members of the United Nations and by neutral countries until January 1, 1947. It will come into force as soon as it has been signed by three further countries provided they sign before the end of 1946.

Specialloid Limited have formed a Canadian subsidiary company—Specialloid (Canada) Limited, with a modern factory at 425, River Street, Verdun 19, Montreal. The factory has been laid out for the production of Specialloid pistons for all types of vehicles operating in the Dominion, including Diesels.

Induction and Dielectric Heating

Applied to Effect Improvements in Production and Quality

The introduction of electronics to industry has brought about many outstanding improvements in production and quality during the past few years. Among the more important of these achievements are those whose effectiveness depends on the use of electronic heat, which may be applied in two forms, induction heating and dielectric heating. Brief reference is made to each form and to their fields of usefulness in industry.

ENGINEERING developments during the past hundred years show a steady infiltration of electrical methods in the field of mechanical engineering, a convergence of the mechanical and electrical sciences and the influence of progress of one branch upon the other. There is also the incidence of significant discoveries leading to the construction of new machines and altered trends of development. Amongst such discoveries in the last fifty years are Thomson's researches providing the quantum nature of the conduction of electricity *in vacuo* or low pressure gases by electrons, Fleming's application of the Edison effect in the invention of the thermoinic valve, and Cooper-Hewitt's development of the mercury-arc rectifier from the mercury-vapour discharge lamp. It is only in the last few years, however, that the fundamental identity of the widely different applications of electrical discharge phenomena in the various branches of engineering has become generally recognised. Evidence of this recognition is provided by the use of the word "electronics" as a generic term covering the study and application of devices embodying the conduction of electricity by free electrons *in vacuo* or by electrons and charged atoms, i.e., ions, in low pressure-vapour spaces.

A wide range of apparatus has been developed by which to apply electronic methods to industry. The range includes small and large high-vacuum valves of the radio type and mercury-arc rectifiers for large powers, as well as a number of other variants such as, the photo-electric cell, the cathode-ray oscillograph the X-ray tube and the gas discharge lamp.

Electrons are directly applied in research and testing, in manufacture, in power control, and in safety devices. They are playing an increasingly important part in all operations in which direct use is made of electricity. Thus, in the manufacturing field, such processes as electrolysis, high frequency heating, arc-welding, resistance welding, electric furnaces, etc., are especially applicable as in each case provision can be made for precise control of the supply of energy. An example of the application of electronics, by means of the oscillator circuit, is electronic heating, which has manifold uses both in the form of eddy-current heating of metals and in dielectric heating of non-conductors. It is in these latter fields of application to which special reference is made in this article.

High Frequency Induction Heating

High frequency induction heating is the transfer of energy from an alternating magnetic field to a particular piece of metal, or section of that piece. The metal may be ferrous or non-ferrous, and the heating takes place as a result of secondary eddy currents being forced to circulate on the surface of the metal itself when it is surrounded by a high-frequency current-carrying conductor. The heat energy in induction heating is a function of the square of the current flowing in the heating coil, a variable power of the frequency of the current, and the resistivity of the material being heated. The eddy-current heating process is used for such purposes as tempering and tipping tool steels, brazing, heating billets for forging, fluxing plated coatings to remove porosity, hardening of the gear teeth and general case-hardening, sintering metallic powders, etc.

The usual form of heating coil consists of a helix of one or more turns of copper conductor surrounding the metal to be heated in such a way that the magnetic lines of force, produced by the coil, interlink with it. The voltage across the terminals of the coil has to be sufficient to cause the proper current to flow through the impedance of the coil, and since this value is a function of the frequency, the terminal voltage will be greater for higher frequency work. Thus the important quantity factors in induction heating are coil current, frequency, and electrical resistivity and permeability. The heating action is comparable to a transformer where the work becomes the secondary and currents are caused to circulate on its surface. The material being heated must be a nominally good electrical conductor.

The use of this method of heating has, for some years, been applied to case-hardening both internal and external surfaces of steel and cast iron components. The purpose of the process is to develop a hard, wear-resisting surface without disturbing the toughness, ductility, machinability and dimensions of the main body. The process is now used in large-scale applications and an automatic control system ensures for each component uniformity of area treated, of depth of hardness penetration, and hardness along the treated surface. By this process the heating of the surface is so fast that the zone under treatment is raised to hardening temperature almost instantaneously—at least before any significant amount of heat penetrates to the main body of the component. The immediate application of a controlled water quench finishes the treatment.

With this process distortion is held to a minimum because only the surface of the component under treatment is heated to critical temperatures. Absence of scaling, spalling, grinding cracks, and of relief of hardening stresses during subsequent machining or grinding, and in many cases the obviating of pre-annealing of pre-normalising treatment, all result from the unusual speed of heating and uniformity of structure obtained.

Dielectric Heating

A new form of electronic heat has recently been developed and applied which is adaptable to non-metallic materials. This is known as "dielectric heating." The two forms, induction and dielectric-heating, are somewhat analogous. The fundamental process was first looked upon as a hindrance in the attainment of high efficiency in electrical apparatus, and shows up as core loss in alternating current motors and transformers, which is simply induction heating, and as dielectric loss in capacitors and high voltage cable, these being dielectric heating in its elemental state. It is an established fact that as a magnetic field surrounds an alternating current-carrying conductor, an electric field is present between electrodes of opposite alternating current potential. Also, any material, whether it be air, liquid, or solid, that is placed between these electrodes will absorb energy from this field.

The heat energy liberated in a unit mass of nominally non-conducting material in an alternating electric field is a function of the square of the voltage impressed on the material, the first power of the frequency of the electric source, the power factor of the material at the particular frequency, and the dielectric constant of the material. With these facts in view, it is of interest to consider the analogies between dielectric heating and induction heating. The loss in dielectric work varies as the square of the voltage applied to the electrodes, while the loss in induction heating varies as the square of the current circulating through the coil. Hence, dielectric is a voltage phenomenon while induction is a current function. The heat produced by dielectric heating varies as the first power of the frequency of the potential applied to the electrodes, while in induction work the heat is a function of the square of the frequency at lower values, dropping off to somewhere near the square root of the frequency at higher values. And last, the heat produced in dielectric heating is a function of the loss factor of the material, obtained by multiplying the power factor by the dielectric constant, while induction heat depends on the resistivity and permeability of the metal being heated. These are the values which should always come to mind when either induction or dielectric heating are being investigated. If the material for dielectric heating has a low loss factor, the frequency must be raised and the voltage increased to compensate for it. If the material is thin, and thereby susceptible to flash-over, the frequency must be increased and the voltage kept below the flash-over value.

The dielectric field is usually produced by flat electrodes, but other forms of electrodes may be used as the source of high frequency potential, depending upon the type of work to which this form of heating is applied.

As the application of dielectric heating is a recent development, it is natural to consider what advantages it offers over conventional heating that will warrant the cost of the necessary equipment and its installation. The most important advantage of dielectric heating is its ability to generate equal amounts of heat in every section of a mass of material regardless of whether this section is at the surface or at the centre. This is extremely important in many heating operations whether drying, as in the case of cores or moulds in the foundry, or for producing physical changes, as in the polymerisation of thermosetting plastics and other organic materials, and in the uniform heating of material for sterilisation. With the conventional heating of materials, the higher temperature is at the surface, whereas with dielectric heating there is an overall heat flow from the centre to the surface. The degree to which this effect manifests itself depends upon the rate of heat input to each unit mass.

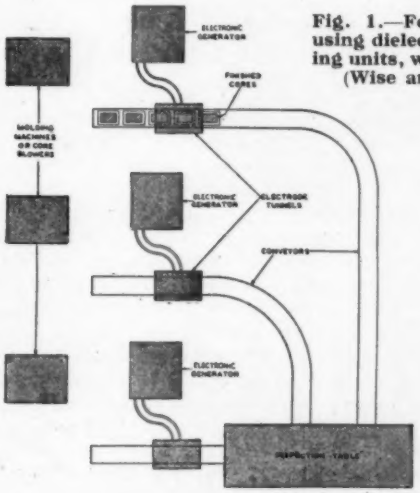

Fig. 1.—Foundry layout using dielectric core baking units, with conveyers. (Wise and Moran.)

Every process that requires the heating of non-conduction material is a potential user of dielectric heating. Probably the most publicised use has been in the heating of thermosetting plastics for polymerisation. This application has been in many individual forms, but predominant has been the pre-heating of preforms for transfer moulding. The conventional method of heating is to use a small electrical oven to raise the temperature to approximately 350° F. at which curing takes place. The preform may range in size from thin wafers to large blocks of material, and as the size goes up, the difficulty to heat it uniformly by oven means goes up as some high power. As the contribution of transfer moulding to the plastics industry has been in the direction of larger products, it can be seen that the whole scheme was greatly limited by the inability to uniformly heat the preforms. The heat set the outside of the preform while the inner portion still remained uncured. Dielectric heating enables the plastics manufacturer to uniformly heat this material, thus speeding up the heating cycle, and at the same time, making the material much more fluid from a moulding standpoint. This latter quality permitted much faster press closing, much less wear and tear on dies, and an all-round superior product. Since dielectric heating is actually intermolecular heating, the improved curing of plastic material is believed to be a result of this. In other words, by working directly on the molecules, an acceleration of the polymerising, which is itself a molecular reaction, takes place.

This form of heating has now been applied to the baking of foundry cores and is proving a solution to many production problems in the foundry. In the first place, it was necessary to develop new thermosetting binders for dielectric baking to replace the oil and cereal binders usually employed in oven-baking cores and moulds. After much research by the Induction

Heating Corporation , New York, in conjunction with foundries, it has been found that, using thermosetting binders, cores can be baked at great speed. Small-sized cores can be baked in as little as thirty seconds, while larger cores take no more than a few minutes for complete baking. The entire process of moulding and baking of cores prior to pouring, which, by ordinary methods, takes hours to perform, is reduced to minutes when dielectric heating equipment is used.

In addition to the time factor, consistently uniform baking of cores is an inherent characteristic of this new foundry process. This development also makes it possible to have line production in the core-making department of any foundry since cores can be conveyed to a specially formed baking tunnel, as shown in Fig. 3. The speed can be adjusted to ensure that each core receives the right amount of heat for thorough baking. There is no heating up time by this method, and the equipment takes up much less space than the conventional ovens. Moreover, the whole process is under control, and there is no possibility of burning or under-baking.

In regard to costs of operation and productive capacities of dielectric heating installations in a typical foundry, Wise and Moran refer to a dielectric baking unit, such as the Induction Heating Corporation's Ther-Monic Model M-700, which is capable of baking approximately 360 lbs. of dry-sand cores per hour. Actual foundry production records show that the power consumption for a unit of this type averages not more than 25 kilowatt-hours per hour ; at a power rate of 1% per kilowatt-hour, the hourly cost of operation would be 25 cents. Larger production rates can easily be estimated from these figures. For example, a plant running 24 hours a day and using four dielectric heating units of this type, having a power output of 12 kilowatts each, would be capable of producing approximately 17 tons of baked dry-sand cores per day at a power cost of only 24 dollars.

Maintenance requirements on these dielectric heating generators are exceptionally low. Power tubes used in this equipment have a life expectancy of from 4,000 to 6,000 hours. When necessary tube replacements can be made quickly by the plant engineer. These dielectric heating generators have proved themselves practical machines for factory use.

It is noteworthy in connection with dielectric and induction heating that Metalectric Furnaces Ltd., Cornwall Road, Smethwick, Birmingham, have recently made an agreement to represent the Induction Heating Corporation, U.S.A., in this country. Thus, information regarding the unit to which Wise and Moran refer is available from Metalectric Furnaces Ltd.

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Tiled Roof Replaced by Sheet Aluminium

An unusual engineering project of the post-war period was the recent completion of the new aluminium roof of the Union Terminal, Cincinnati. The aluminium portion includes the roofing of the dome-shaped area and two arched roofs covering the main body of the terminal, as well as three parapet walls extending to the front façade. A total of 32,000 sq. ft. of roofing was involved. Various available roofing materials were studied, and a decision made in favour of aluminium as being simple to prefabricate and easy to erect. In addition to meeting all requirements of weather resistance and low maintenance costs, such a roof is extremely light in weight, weighing approximately one-twentieth of that of the old tile roof which it replaced.

With the exception of the flat areas, where $\frac{1}{8}$-in. aluminium sheet was used, the entire dome-shaped roof, barrels and parapet walls were covered with 14-gauge aluminium sheet, of which a total of 1,123 sections were used. Because of the contour of the dome-shaped area, it was necessary for each section of sheet to be tapered to insure precise fitting as the sections converged toward the top. In addition to the preformed aluminium field sheets, approximately 11,500 ft. of extruded alumnium batten and cap were used.

Prior to installation of the aluminium roof, it was necessary to remove the old tile roof and to prepare the underlying structure for the new surface. The concrete forming the base for the old roofing was left in place as was the 30-lb. layer of felt. Repairs to the existing felt layer were made where necessary. Over this a 1-in. layer of cork was mopped on, followed by another layer of 30-lb. felt. Over the last layer of felt a layer of 15-lb. asbestos felt was placed with the outer surface dry. This 1-in. layer of cork develops approximately the same value of insulation as the previous roofing developed. All bolts and washers were made of aluminium utilising in each case the alloy best suited to meet the requirements of the job in hand.

The completed roof and parapet walls present a compact network of interlocking sliding aluminium joints capable of withstanding any conceivable amount of expansion and contraction. All parts of the roof are readily accessible in the event that emergency repairs are necessary.

Control of Aluminium and Light Alloys

Government wartime control of the purchase of aluminium and light alloys has been removed with effect from August 1, under two Orders made by the Ministry of Supply,—the Control of Aluminium (No. 7) Order of 1946, and the Light Metals and Alloys Fabrication (No. 2) (Revocation) Order, 1946.

As the Ministry has made a contract for the supply of 215,000 metric tons of virgin aluminium from Canada during the years 1946-1947 (announced in an earlier issue), it will continue to be the sole buyer and seller of virgin aluminium.

The Ministry is also cancelling directions previously issued to the Light Alloy Industry requiring the segregation of light-alloy scrap into various categories, and the making of statistical returns. Arrangements have been made for statistics to be furnished on a voluntary basis by the industry, and totals will be issued monthly by the Ministry of Supply for publication in the trade press.

Copies of the above Orders can be obtained from H.M. Stationery Office, York House, Kingsway, London, W.C. 2, or through any bookseller, price 1d. each. (S.R. & O. 1946. No. 1269 and 1270.)

Cutting Fluids in Machining Operations

By L. Massey, M.Sc., and A. A. Pollitt, M.Sc., F.R.I.C.

Research Department, Metropolitan-Vickers Electrical Co. Ltd.

There is considerable divergence of opinion and a lack of agreement concerning the application of cutting fluid, yet the yearly consumption of undiluted cutting fluids in Great Britain alone is reckoned in millions of gallons. It is difficult to conceive that an industry will use this material unless it has definite evidence that either economies in production or improvement in the quality of the product result. The authors reviewed and discussed the types, properties, application and examination of cutting fluids in a paper before the Crewe, Liverpool and Manchester Sections of the Institution of Production Engineers, the main features of which are given in this article. The authors direct attention to many important aspects of the subject, the significance of which is not always fully appreciated in practice.

Introduction

IN making a review of the subject of cutting fluids, the first impression received is one of a lack of agreement concerning their practical application. Divergence of opinion exists regarding the type of fluid necessary for a specific machining operation, or for which operations a specific fluid may be used. The question even as to whether a cutting fluid need be used or not is often the subject of argument.

This state of affairs may be illustrated by citing one particular instance drawn from a survey of cutting practice made by McKee[1] for the A.S.M.E. :—Out of 63 industrial plants concerned with the turning of low carbon steel, 27 were using water emulsions, 19 were using neat oils and 17 were not using any cutting fluid.

Such divergence of common practice should not be possible if a field is covered by well-established principles and attention was turned therefore to the theory of metal cutting and the function of the cutting fluid. It was found that the precise mechanism of cutting is not yet fully established and that the function of the cutting fluid, except as regards its action as a coolant, is still often a matter for conjecture. This is perhaps not surprising because of the complexity of the problems involved, particularly those concerned with boundary lubrication.

The lack of guiding knowledge as to mechanism is the direct cause of the present inconsistencies in practice. Tests made in the shops under one set of conditions embracing work material, tool material, shape and setting, speed of cutting and so on, can give only indications for those specific conditions and are useless as a basis for generalisation. The cutting-oil technologist, on the other hand, working from a knowledge of the chemical and physical characteristics of each type of cutting fluid, can give only general guidance regarding application because there are so many variables in cutting conditions in practice, each of which may call for a specific combination of properties in a fluid and/or for a specific cutting technique.

Further to these very real difficulties, personal preference and prejudice based on very meagre evidence causes much confusion. The skilled operative is apt to hold very strong opinions regarding the virtues or otherwise of particular fluids, and in the absence of well-established principles it is difficult to correct wrong conclusions which have been formed without a full appreciation of all the variables concerned. It is also clear that sales propaganda is unhampered when no positive knowledge exists.

1 McKee, *Trans. A.S.M.E.*, 1930, Vol. 52, p. 103.

From the confusion engendered by these preliminary findings, we are rescued by one concrete fact—consumption. The yearly consumption of undiluted cutting fluids in Great Britain alone is to be reckoned in millions of gallons and it is impossible to conceive that the engineering industry is purchasing so much material unless it has definite evidence either that economies in production or improvement in the quality of the product is a result.

It will be agreed generally that the engineer uses cutting fluids for four main reasons :—

1. To achieve an improved surface or finish to the work.
2. To prolong the life of the tool.
3. To increase cutting speed.
4. To flush chips away from the work surface.

In considering how cutting fluids enable these objectives to be attained in greater or less measure, it is convenient to discuss the subject under the following headings :—

Mechanism of cutting.
Properties desirable in cutting fluids.
Types of cutting fluids commercially available.
Chemical examination : (*a*) To evaluate the usefulness of a fluid.
(*b*) To maintain the quality of accepted fluids.
Application of cutting fluids.

Mechanism of Cutting

The mechanism of cutting has been investigated by a number of workers, and it is possible to form a broad mental picture of the cutting process. The physical phenomena involved are compression, plastic flow, shearing and wedging. The mechanisms discussed apply to all cutting edges, irrespective of their nature, from the rigidly held, carefully shaped and positioned planer tool to the irregular edged abrasive grains on a wheel or sheet of emery paper.

Two types of turning or chip can usually be recognised. Considering first the case of a brittle metal, when the tool, ABC, approaches the work (Fig. 1*a*), the cutting edge first compresses the metal until crumbling occurs at the point B. As the tool advances, great compressive stresses are set up, probably with very slight plastic flow, until ultimately the metal shears along the line BI and the chip BGHI is detached at the shear angle θ (Fig. 1*b*). The sequence is repeated resulting in the formation of the discontinuous type of chip.

If the material is ductile, the compressive stresses cause plastic flow of the metal towards the free space above the cutting edge—Fig. 2*a*. If the compressive stresses do not exceed the shear strength of the metal the flow continues and the material escapes upwards along the face of the tool, forming the continuous type of chip—Fig. 2*b*.

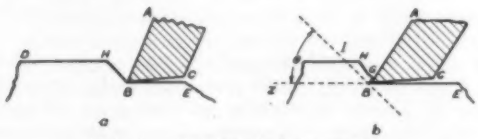

Fig. 1.—Cutting brittle metal.

Very tough material may develop a crack opposite the nose of the tool and in this case the action of the tool resembles that of a wedge with the crack preceding the nose. The metal is thus torn apart at B—Fig. 2*c*.

The continuous chip is often associated with a built-up edge on the nose of the tool. Ernst[2] and his colleagues believe the formation of this built-up edge to be due to the existence of a very high coefficient of friction between the tool face and the chip face although high pressure and temperature may also play a part. In general engineering practice the coefficient of friction between unlubricated metal surfaces is of the order of from 0·1–0·3, but it has been shown that all normal surfaces have adsorbed surface films which function as boundary lubricants. If metal surfaces are made chemically clean the coefficient of friction becomes extremely high and the "stick" or cohesion between them becomes so great that if relative movement is caused, shearing and tearing of the metal occurs. It is also known that metal surfaces, to which relative movement is imparted, do not slide continuously. They "stick" and "slip" alternately, the process being rapidly repeated. This "stick-slip" action is more marked as the coefficient of friction increases.

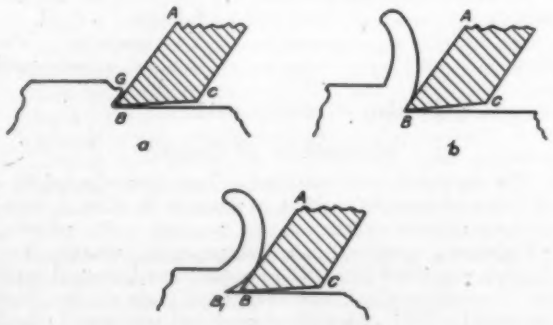

Fig. 2.—Cutting ductile and tough material.

The chemically clean surfaces exposed during metal cutting are likely to have high coefficients of friction and during the "stick" interval of time, the adhesion between chip and tool is so great that as the tool moves forward shear takes place along the line DE—Fig. 3*a*, and a loose triangular piece of metal is formed attached to the work at the base. With continued forward motion of the tool this piece must also shear from the work and the result is the formation of a step on the machined surface of the type shown at *x* and *y* in Fig. 3*b*.

Ernst attaches great importance to this mechanism and has shown in a series of very fine microphotographs that the fragment of the built-up edge which remains on the work surface as a step is the cause of the transverse

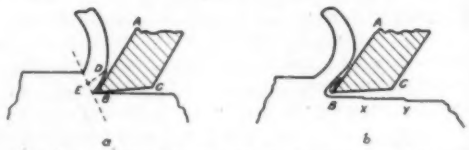

Fig. 3.—The formation of steps on a machined surface

irregularities which form the major type of blemish on machined surfaces.

Whether the theory advanced by Ernst is correct, or whether it may have to be modified in the light of other theories being explored, the facts still remain that the presence of a built-up edge is accompanied by the presence of steps on the surface of the work, of the type shown and that in the presence of a lubricant and slow speed the steps are not produced.

It is believed that this very brief resume covers all the fundamental processes involved in cutting and that from it an understanding of the desiderata and behaviour of cutting fluids can be gained.

The subject can be treated mathematically, of course, and many strictly engineering aspects exist such as the problems of vibration and chatter patterns, but it is considered that these are not relevant to the present subject.

Properties Desirable in Cutting Fluids

A study of the mechanism of cutting has so far revealed only two ways in which a cutting fluid can function—namely, as a coolant and as a lubricant.

The need for a coolant is self-evident. Mechanical energy expended in deforming and shearing the metal must appear in the form of heat and whatever variations or modifications of technique are employed this inherent effect of cutting—*i.e.*, evolution of heat—is always present. Friction which occurs between the nose of the tool and the work or between the face of the tool and the chip is a further cause of the development of heat, but in this case the heat is not a fundamental quantity bearing a fixed relation to the true cutting operation. Frictional heat varies according to the relative incidence of a number of factors not yet thoroughly evaluated.

Whatever the origin of the heat, it is usually desirable to secure its dissipation from the tool and the work. If the tool gets unduly hot its hardness and therefore its life will be reduced, whilst if the work is not kept cool it may be machined in a distorted or expanded condition resulting in inaccurate final dimensions. In some cases it may even be necessary to pack the work with solid carbon dioxide whilst machining is in progress in order that the dimensional limits may be accurate over the range of temperature through which the product is intended to work.

The characteristics required of a coolant are: (1) Low viscosity to facilitate maximum heat dispersion by convection currents; (2) good wetting power to ensure good and uniform thermal contact between coolant and metal; (3) high specific heat or thermal

2 Ernst and Merchant, *Chip formation, friction and finish*. Published by Cincinnati Milling Machine Co.

capacity ; (4) a high heat conductivity to facilitate heat dissipation by conduction ; and (5) a high latent heat of vaporisation. Of these, low viscosity and good wetting power are probably the most important. Small variations in the other characteristics can be overshadowed by the latitude possible in the rate of flow of the coolant, but high viscosity or poor wetting power are likely to detract seriously from the efficiency of the fluid as a coolant. The value of a high latent heat is doubtful unless one presupposes that the tool is to operate at the boiling point of the fluid.

The considerations involved in the function of the fluid as a lubricant are not so obvious and it is even disputed sometimes that the fluid does so function.

Let it be made clear, therefore, that at least under some conditions, lubrication is a function of the fluid by quoting the work of Boston[3] and his colleagues. They carried out machining tests using a number of fluids and arrived at the general conclusion that with the exception of brass the use of a cutting fluid resulted in an economy of power. In one particular case, that of drilling S.A.E. 1020 steel using a sulphurised oil, the drilling torque was reduced by 33% as compared with drilling dry. When it is remembered that a large proportion of the power input must be absorbed in cutting, it will appear obvious that the reduction of frictional losses must have been great.

The two surfaces where lubrication most probably plays a part are between the tool nose and the work and between the tool face and the chip. At a first glance a simple conception seems possible—namely, that the problem is merely one of reducing the coefficient of friction and thereby the power absorbed and the heat developed. It has been shown, however, that lubrication affects the quality of the finish and moreover, a little further thought raises two very difficult questions : (1) How does the lubricant penetrate to the rubbing surfaces ; and (2) how can any lubricant resist the enormous pressures known to exist at the cutting edge, which pressures are far in excess of the recorded film strengths of any lubricants ?

The only answer to the first question seems to be capillary action. However carefully machining is carried out, the resultant surface is never perfectly flat but has its system of "hills" and "valleys." Mated surfaces do not touch therefore, at all points, but only at the "high spots." This creates a network of channels and passages which a fluid can penetrate by capillary action. If a crack precedes the tool nose, this also is a channel of access for the fluid. This explanation, accepted tentatively for want of a better one, applies mainly to the continuous type of chip which slides over the tool face. In the case of a brittle metal and discontinuous chips, there is little movement of the chip relative to the tool face because the action is one of crushing, and lubrication therefore is not necessary.

It is difficult to visualise how lubrication at the extreme nose of the tool is helped by capillary action, but that such action can take place along the face of the tool at the point where heavy wear usually occurs is readily appreciated. At this point, factors such as poor mating caused by jumping, irregular stresses, vibration and distorted curling of the chip may open up gaps of appreciable dimensions between tool and chip.

One of the main objections to the capillary flow theory is that there may be no time for this to take place, and it is of interest to note in this connection that there is general agreement that with the very high-cutting speeds possible with sintered carbide tools, no lubrication effects are observed, and it is accepted that in this case the fluid functions solely as a coolant.

It is possible that volatile products liberated from the cutting fluid by the high temperature at the tool face may play an important part in providing boundary lubrication at this point. Such products are likely to be gaseous and therefore will be very rapidly absorbed into the capillary systems and there they may bridge the gap between higher and lower cutting speeds at which latter it is generally agreed that capillarity has time to operate, whereas at the high speeds it is believed that no film lubrication is present. The work of Brownsdon[4] on the effect of added substances on the lubricating properties of oils indicates that products of decomposition can function as boundary lubricants.

If it be accepted that lubricant does find its way to the rubbing surfaces, the next question arising is, how does it resist the high pressures ? Practical experience shows that it does function in spite of the fact that the calculated contact pressures (*i.e.*, circa 100,000 lb./sq. in.) are far in excess of the maximum recorded film strengths of any lubricant as measured on machines such as the Timken apparatus. It must be admitted, however, that our knowledge of the mechanism of boundary lubrication and of film strengths is far from complete. The important point is that in practice the order of merit of a lubricant for severe cutting duty corresponds roughly with the general trend of film strength ; the higher the film strength the better the cutting performance.

Reference has already been made to the opinion of Ernst, that most of the surface faults on the work are transverse irregularities which are steps left by fragments of the built-up edge and that the built-up edge is the result of a high coefficient of friction. The presence of a lubricant reduces this coefficient between tool and chip and makes impossible the high cohesion which shears off the triangular piece. Lubrication should eliminate the built-up edge, therefore, and result in a smooth (or smoother) machined surface. Ernst presents experimental evidence in support of this theory in the shape of microphotographs of planing cuts on steel. The face of the tool waiting to commence the cut will normally carry a film of adsorbed low film strength material and at the beginning of the cut the coefficient of friction will be relatively low ; no built-up edge is present and a smooth surface results. As the cut continues, the freshly formed surfaces passing over the tool rob it of adsorbed lubricant and the coefficient of friction rises, with signs of a rough surface appearing. Finally, the friction becomes sufficiently high to cause the formation of a built-up edge and marked surface defects appear.

Ernst repeated the experiment under exactly similar conditions except that an efficient cutting fluid—carbon tetrachloride—was used, and showed, that the lubricated work was free from the surface defects associated with dry cutting. The speed of cutting used was extremely slow but the results are of great interest and cannot lightly be dismissed.

It is now possible to make a tentative statement regarding the properties to be desired in a cutting lubricant, but just as the function of a lubricant is more difficult to define than that of a coolant, so is the specification of lubricating properties more difficult than that of coolant properties.

3 Boston and Oxford, *Trans. A.S.M.E.*, 1933, Vol. 55, p. 1.

4 Brownsdon, *J.M.E.* General discussion on lubrication, 1937, Vol. 2, p. 254.

The lubricant must have boundary lubrication properties; that is, it must be of the type which can modify the coefficient of friction between two surfaces between which no fluid film exists. In this condition the friction is probably made up partly by the surface forces of attraction between the two materials and partly by the work required to lift and slide the high spots of one surface over the high spots of the other, or to shear the high spots, if the force normal to the surface is too great to permit lifting of the one over the other. Lubricants of this type exist as films physically or chemically attached to the metal surface and having a film thickness of from one to ten molecules. They are frequently characterised by the presence of molecules which possess a carboxyl group linked to a long hydrocarbon chain, the carboxyl groups attaching themselves to the metal so that the hydrocarbon chain stands up from the metal surface as does the pile from a carpet.

Because of the high pressures involved, the lubricant should belong to the class known as extreme pressure lubricants, that is, it must attach itself so firmly to the metal surface that it is not squeezed off. It must also be chemically stable at high temperatures because it has been shown that extremely high temperatures are developed over the slip portion of the "stick-slip" cycle. Alternatively, any products of decomposition at high temperatures must themselves be boundary or extreme pressure lubricants.

The lubricant must have low viscosity and high wetting power in order that capillary spaces between tool and work may fill as rapidly as possible. Both these properties are desirable in a coolant for reasons already given and fortunately there is nothing in the specification of a lubricant which is incompatible with the major requirements of a coolant.

In addition to the foregoing primary requirements, cutting fluids must possess also certain secondary properties. They must not cause or accelerate metallic corrosion or both work and machine will suffer. They must be non-toxic and non-irritant to the skin. They must be stable under storage and operating conditions and in particular they must neither gum nor separate out solid constituents. They should possess high flash point so as to minimise fire risks and finally it may be added that they must be cheap in price.

Commercially Available Cutting Fluids

The development of cutting fluids demands an appreciation of the properties already outlined and a knowledge of the materials commercially available for their formulation. It is obviously economically desirable first to select liquids which are in cheap and plentiful supply and which possess initially and inherently as many of the desired properties as possible, and then to blend, modify, make additions and generally chemically process these basic materials until suitable final products are obtained.

A survey of available materials gives an unhesitating answer as to the most suitable base materials. Water is the best coolant and oils are the best lubricants. Neither material possesses a high performance in both respects and as a result, cutting fluids are divided into two classes; those based on water and used where coolant properties are of prime importance and those based on oils and used where lubrication is of prime importance.

Considering the use of water alone as a coolant, three obvious disabilities are apparent:—Its wetting property is poor, its lubricating power is small and it causes corrosion of ferrous materials. In the early days of cutting fluids, soda was added to water to minimise its corrosive effects, and whilst this practice was purely empirical at the time, it is now known that if an alkalinity not less than pH 9·2 is maintained in water, corrosion is inhibited. The soda solution still had poor wetting power and no lubricating value and the next step was the addition of soap. By thus lowering the surface tension, the wetting power of the fluid was improved and some slight lubricating property was imparted. Further development came with the incorporation of fatty oil, the object being to leave, after evaporation of the water, an oily film on the metal which would be protective against corrosion. These fluids were produced by adding caustic soda to a fatty oil, such as lard-oil, in quantity insufficient to effect complete saponification. The result consisted of a mixture of water, soap and fatty oil in which the soap acted as an emulsifier keeping the unsaponified oil in suspension in the form of droplets. These preparations, made up in the engineering shops were not very satisfactory because the emulsions were frequently unstable, became rancid, were not very rust-inhibitive and were definitely corrosive towards non-ferrous metals.

Realising the potential field for a specialised product, the oil industry then began to market cutting pastes which could be transformed into cutting fluids by mixing with water. It would appear that the first use of mineral oils was made in these pastes. They consisted of a dispersion of mineral oil in water, using soaps as emulsifiers, and were fairly successful in application. The mineral oil, though not so good a lubricant as the fatty oils, still possessed lubricating properties adequate for the lighter duties and the film left on evaporation was definitely superior to that of fatty oil in the protection against corrosion, being neutral also towards brass and copper and causing no greening.

The pastes were difficult to break up and were gradually replaced by the soluble oils of types similar to those used to-day and which are easily diluted with water. The soluble oils, which comprise the major proportion of present-day cutting fluid consumption, are essentially an emulsion of mineral oil in water, the dispersion being effected by the presence of an emulsifying agent. Development and improvement have been associated mainly with the nature of the emulsifier or with the addition of small amounts of compounds intended to increase the oiliness or extreme pressure lubricating properties. The first emulsifiers were the soaps produced by the saponification of fatty oil. These were followed by rosin soaps, but in both cases their use was associated with such disabilities as rancidity, development of gummy residues, oil separation and poor dispersion of the mineral oil. Next to come into prominence were the sulphonated oils and these have retained their importance up to the present time. These sulphonated oils, which are prepared by treating vegetable oils, in particular castor oil, with sulphuric acid, must not be confused with the sulphurised oils although they contain sulphur. Most of the soluble oils now marketed contain sulphonated castor oil in the form either of the sodium or the ammonium soaps.

Other emulsifiers now in use include petroleum sulphonates, diethylene glycol, sodium naphthenate, sulphonated alcohols and other synthetic products developed specifically for their properties as surface tension depressants.

The degree of the dispersion of the mineral oils in water is a function of the efficiency of these emulsifiers and this can generally be assessed from a microscopic examination of the water solution. In a poor emulsion the oil globules are relatively large and unevenly spaced in the aqueous phase, whereas, in a satisfactory emulsion the oil globules are small and uniformly dispersed. Dilution of these fluids with water yields a liquid having a milky appearance due to the scattering of light by the discrete oil globules.

In unfavourable circumstances, or if the emulsifier is of poor quality, the oil globules may coalesce and separation of oil from water then occurs. The protection of steel against corrosion following machining operations is usually ascribed to the residual oil film left after evaporation of the water, but it may equally well be due to the deposition of oil from an unstable emulsion or from the cracking of an emulsion due to the temperatures coincident with machining. Emulsions of this type can be very temperamental with respect to their stability and high efficiency dispersants have recently been developed by the use of which the mineral oil is so dispersed that individual globules approach colloidal dimensions, and the resulting emulsions are water-clear and possess great stability. Very favourable comment has been made upon some of these clear emulsions which, in addition to possessing greater stability appear to be more rust-inhibitive than the milky type. Though some observers have reported that gummy residues result from the use of these clear emulsions, it is possible that this may have been due to the use of excessive quantities of fatty base emulsifier, added in an effort to produce clear fluids, whereas the best practice is to use only small quantities of non-oxidisable high efficiency emulsifiers. This would accord with the accepted fact that too high a proportion of fatty matter, whether present as oil, soap, fatty acid or sulphonate, will cause gumminess due to oxidation.

In parallel with improvements achieved in recent years in emulsification, improvement in lubricating value has been sought. The most obvious step was to supplement the known deficiency in the boundary lubrication properties of mineral oil by the addition of fatty oils which have always been accepted to possess more oiliness than mineral oil. Instead of fatty oil, the free fatty acids obtained from the oil are frequently used and in practice all possible combinations of fatty oil, fatty acid, soap and caustic soda are to be found. As regards the nature of the fatty component, lard, rape, sperm, castor, tallow and other fixed oils and fats may be present, usually singly but sometimes in combination.

In addition to the main constituents there may be present small quantities of other compounds, added for special purposes, such as alcohols, petroleum and coal tar hydrocarbons to aid in blending, chlorine or sulphur compounds to improve lubrication, and antiseptics to restrict bacterial growth and so minimise skin infection. The most common composition found to-day is the milky mineral oil-water mixture, emulsified with sulphonated castor oil and containing some proportion of lard-oil or other fatty base material and a small proportion of antiseptic.

It must be emphasised, however, that the success of these materials is not only a matter of formulation, it depends also and very largely on the art of mixing and blending. Manufacturers are very reticent on these points and it is not possible to more than generalise on the subject.

Water soluble cutting oils are used where coolant properties are of primary importance. At the other end of the scale there is the class of oils used where lubrication is essential, as in heavy duty. Lard-oil was the original oil used for this latter application. It is still used and is highly esteemed by some engineers. Its useful property is its oiliness ; its chief defects are high price, tendency to turn rancid, smell when heated, tendency to gum and corrosive effect on non-ferrous metals. Other fatty oils have been and still are used but in general they share the good and bad points of lard oil, and there appears to be no question that they give a high performance in severe duty application requiring a high degree of oiliness. Their defects led to the introduction of mineral oils and whilst these lack the boundary lubrication properties of fatty oils they yet possess useful lubricating value and they do not suffer from many of the drawbacks of the fixed oils ; they do not turn rancid, corrode metals, gum, develop objectionable smell and they are cheaper than fatty oils—a most important point. For these reasons they have found wide use in work requiring a medium lubricant or in work requiring a coolant but on which the use of the coolant emulsion is not desirable—*e.g.*, brass.

It was then but a small step to the next development, the blending of mineral with vegetable oils. Apart from the obvious motive of decreasing the cost of fatty oils by diluting with the cheaper mineral oil until the algebraic mean of their lubricating power just equalled the severity of the load, which presumably was the initial basis on which blending depended, other possibilities were involved. The presence of the mineral oil could materially reduce the corrosive influence on metals, greater chemical stability would be conferred, gumming would be reduced and the lower cost would make possible a higher rate of circulation so increasing the coolant action, the whole resulting in a far better balance between lubrication and cooling than was possible with emulsions of fixed oils alone. These possibilities have been realised in practice and compounded oils consisting of varying proportions of fatty oil in mineral oil are widely used and are known as mineral-lard-oil irrespective of the identity of the fatty oil and contain from 10–40% of the latter. They are graded in value according to the proportion of fatty oil present and are frequently diluted further in the shop with mineral oil.

It is, however, pertinent to consider to what extent increase in fatty oil above a certain minimum affects the performance. Bearing in mind the work of Southcombe and Wells showing the enormous effect of adding small percentages (of the order of 1%) of fatty acids to mineral oils, it would seem likely that if a mineral oil contains the small quantity of fatty oil necessary to form and maintain a layer of polar molecules on the metal surface to provide for boundary lubrication, further addition of fatty oil would serve no useful purpose. It is still the practice to increase the fatty oil content according to the severity of the machining operation but it may be wondered whether the practice is based solely on algebraic reasoning, though it may be conceded that this type of reasoning is the only sound basis upon which the man in the shops can operate, because the accurate measurement of relative values involves control of so

many variables that even the specialist in this field can be led astray.

Natural fats are now sometimes replaced or supplemented by synthetic fats but the considerations involved remain essentially the same.

About 20 years ago the sulphurised oils were introduced. The value of sulphur as a lubricant had been recognised for some time and practical experiment had shown that if elemental sulphur was dissolved in fatty oil a cutting medium was obtained which was superior in performance to the original oil. It is now known that sulphur imparts extreme pressure characteristics, but its original use did not arise from this knowledge but from practical experience. The sulphur was cooked into the oil to produce a sulphurised fatty base containing up to 20% of sulphur and this was then diluted with mineral oil to a suitable working strength, containing about, say, 1% of sulphur. It was an obvious step then to add sulphur to mineral oils and in this case the improvement of cutting performance was even more marked than with the sulphurised fatty oils. Various chemical and physical processes for incorporating sulphur are practiced—*e.g.*, the sulphur may be thermally cooked into the oil in the elemental form or it may be added in the form of an oil soluble sulphur compound. Another process gives a purely physical dispersion of colloidal sulphur and claims an oil of lighter colour coupled with improved performance.[11] Whatever the process of incorporation, or whatever the form of the incorporated sulphur, it is certain that the sulphur must be in a form capable of reacting chemically with metal, presumably to form sulphide films. At one time attention was directed to petroleum crudes of high sulphur content with the idea that these naturally occurring oils could be refined to yield high sulphur products suitable for use directly as cutting oils. It was found, however, that

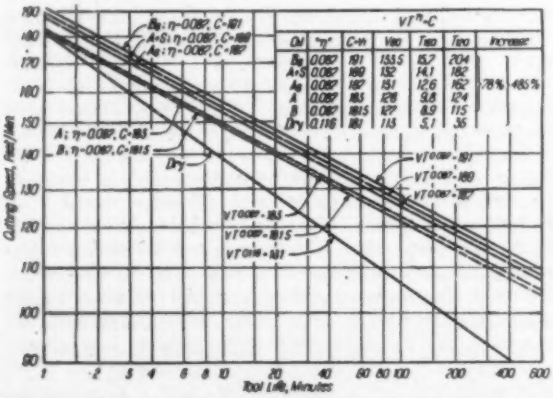

Fig. 4.—Cutting speed tool-life tests for five oils with and without added sulphur when turning annealed and normalised S.A.E. 3140 steel with tools ¾ in. square of high speed steel ground to a shape of 8, 14, 6, 6, 6, 15, 3/64. The depth of cut was 0·100 in. and the feed 0·0125 in. The curves are plotted on modified log-log co-ordinate, the vertical being six times the horizontal.

their performance was no better than that of the low sulphur crudes and the reason is no doubt due to the fact that the sulphur as naturally occurring, is in a form too chemically stable to react with metal during the machining operation.

The sulphurised oils comprise (1) vegetable, animal or mineral oils containing up to about 3% of sulphur and used as bought, and (2) sulphur base oils containing up to 20% of sulphur and which are diluted as required for use with mineral oil to the desired strength. These latter may be a fatty oil or a blend of fatty and mineral oil.

Sulpho-chlorinated oils are also now available (at least in the U.S.A.) and are produced either by dissolving sulphur chloride in oil or by chlorinating a sulphurised oil. They are characterised by good transparency and colour, and a very high performance as cutting lubricants which surpasses that of the straight sulphurised oils.

Sulphurised mineral oils of one type or another are very widely used because their lubricant properties are superior to those of the best fatty oils and their cost is much lower. Their performance has been studied by Boston and Zimmer[5] who have established quantitatively the claims to superiority by carefully controlled drilling and turning tests on various steels using cutting speed and tool life as indices to performance. In these tests, as in the other tests carried out by Boston and his colleagues, the basic equation used has been :—

$$VT^n = C$$

where :—V = cutting speed in ft./min.
T = tool life in mins.
n = slope of line from the horizontal.
C = height or vertical displacement of line.

In Fig. 4, which is reproduced from A.S.M.E., Vol. 30, 1942, p. 793, are given the curves obtained on the following oils :—

(*a*) Dry cutting.
(*b*) Oil A containing 0·1% natural sulphur.
(*c*) Oil B ,, 2·03% ,, ,,
(*d*) Oil A + 0·8% elemental sulphur.
(*e*) Oil B + 0·8% ,, ,,
(*f*) Oil A + 1·6% oil soluble synthetic sulphur compound.

It is not possible to discuss here in detail all the implications of the results of an investigation of this kind but as a typical instance of the superiority of the sulphurised oils a reference to the figure will show that the addition of sulphur to oil B improves the tool life at a cutting speed of 120 ft./min. by 78%. Of the attention now being given to extreme pressure lubrication, the cutting fluid aspect is receiving its share, but in practice progress was made before this theory was much discussed, for the sulphurised and chlorinated oils fall into the class of extreme pressure lubricants.

From time to time other additives have been proposed, and of these graphite is a well-known example. It will be clear that graphite can persist on surfaces when oils would be thermally decomposed and its well-known leafing or "fish scaling" effect will give lubrication when other materials may have been expelled by high pressure. The $VT^n = C$ curve for graphite dispersed in oil has been determined by Boston, Gilbert and Kraus[6] who found that the presence of graphite in an optimum concentration of 0·153% resulted in an increase in cutting speed of 20% for a given tool life or in an increase of from 400–600% in tool life for a given cutting speed. This finding does not appear to have been commercially exploited on account of practical difficulties.

Possibly one of the most likely fields for further development of cutting fluids is the improvement of extreme pressure properties.

To be Continued

11 *Aut. Eng.*, 1942, Vol. 32, p. 29.

5 Boston and Zimmer, *Trans. A.S.M.E.*, 1942, Vol. 30, p. 793.

ALTHOUGH it brings up again a topic which we have discussed several times recently in these columns, we feel that we must refer to a letter recently received from a practising microchemist in this country. Many of our chemists, of which number he is one, have been faced with the problem of equipping new laboratories, either to replace those destroyed during the war, or to cope with the extended programme of scientific research which, we are everywhere assured in print, is desirable, and indeed, necessary for the survival of our nation. To quote from the letter mentioned, he says, "I am still suffering from lack of microchemical equipment. It is deplorable that at the crucial stage in the development of microchemistry in this country the majority of the apparatus is going for export." If the apparatus is indeed going for export, then, much as we welcome support for the export drive, we agree with him. There is a fundamental difference between exporting the fruits of research, and handing out the tools of research. If we persist on the latter line, we shall soon have nothing to export. But the complaints to which we referred some months ago, that Continentals cannot obtain British microchemical apparatus, raise some doubts in our minds. We begin to have a suspicion that British manufacturers of chemical apparatus, misled by a confusion in terms, regard microchemists as small fry. Their apparatus may be small, and the samples on which they work microscopic. But in the scientific scheme of things no one should be in doubt that they bulk large, and they will—if not smothered—continue to increase.

Problems Concerning the Microchemical Balance

Part III.—Construction

By Cecil L. Wilson

The types of microchemical balance which are available in this country are naturally limited to some extent by the war. However, it is undoubtedly true that this state of affairs will not be permanent, and therefore it may be of advantage to consider briefly the different instruments which have been applied to the weighing of small amounts of material.

THE first microchemical balance, as stated in the first article in this series[1] was that evolved by Warburg and Ihmori.[2] This balance was used by them to weigh the water film on a glass surface, and had a very limited application.

It seemed to early workers with small amounts of material that the use of the torsion principle offered advantages for the high sensitivity necessary, since methods of making knife edges did not seem to be capable of the refinements demanded, if the sensitivity of the ordinary beam balance were to reach the desired degree of perfection. Consequently Salvioni,[3] by 1901, had developed a torsion balance capable of carrying a load of 100 mg., and with a precision of 0·001 mg. This balance functioned by measurement, with an eyepiece, of the torsion on a fine wire. Quartz fibres were next used for this purpose, and the most famous example of such a balance is the Nernst balance,[4] designed in 1902.

Quartz-fibre torsion balances were very widely used for many of the early microchemical quantitative determinations. In the investigation of atomic weights of the rare earths and other elements, and in the determination of minute amounts of mercury electrolytically, we have examples of the types of problem which were solved successfully using the quartz-fibre torsion principle. Above all, Emich recognised the possibilities in this small scale work, and made many determinations, both inorganic and organic, which were highly successful.

However, the limitation of the balance working on this principle was that, for the desired sensitivity, the load was extremely small, and therefore vessels to be weighed had themselves to have almost negligible size. Consequently the technique to be applied to such work was, in general, outside the abilities of any but the most outstanding manipulators.

In 1910 Pregl, who was at that time investigating the constitution of the bile acids, required elementary analysis on several milligrams of material. Since Emich's work, more especially in the organic field, had

1 Cecil L. Wilson, *Metallurgia*, 1944, **31**, 101.
2 Warburg and Ihmori, *Ann. Phys. Chem.*, 1886, **27**, 481.
3 Salvioni, *Z. anal. Chem.*, 1904, **43**, 633.
4 Nernst, *Z. Elektrochem.*, 1903, **32**, 622 969.

been so successful, Pregl felt that it should be possible to obtain sufficient accuracy on his limited samples, always provided that it was possible to overcome the apparatus difficulty. In the determination of carbon and hydrogen it would manifestly require revolutionary developments in one of two directions. Either the absorption tubes must be reduced in weight to a point where they could be dealt with on the existing sensitive balances, or some balance capable of taking very much greater loads than were at that time recognised to be possible, must be devised. Pregl decided that while absorption tubes could be somewhat reduced in size, the gain to be expected in that direction was limited, and that the second approach was therefore the one on which to concentrate.

Kuhlmann, at that time, manufactured a short-beam assay balance with high precision. Owing to the short beam, it had the advantages to be derived from a light beam which was, in addition, free from large errors due to unequal expansion, or bending of the beam arms, and from a short oscillation period. Emich had, by 1910, made some experiments with this balance, and had found it suitable for inorganic determinations, although it was still not capable of taking the mass required by the modified absorption tubes visualised by Pregl.

Pregl, therefore, asked Kuhlmann to attempt, by improved methods of grinding the knife-edges, and close attention to similar details of construction, to produce a balance for heavier loads. The result of this was the original 19-b balance with a special rider, and fitted with hooks to carry absorption tubes. Using this balance, Pregl was able to carry out elementary analysis for carbon and hydrogen on 8 mg. of material.

By further improvements in construction to give a standard balance capable of carrying a load of 20 g., and with a sensitivity of 0·001 mg., and by improvements in technique, Pregl, as is now well known, was able to reduce the amount of sample to 2 mg. in 1912.

Emich, quick to see the advantages in this type of balance, also switched to its use. The larger vessels now permitted extended the inorganic applications of microchemistry very widely.

The devising of the Kuhlmann microchemical balance may be held to have set the direction of further developments in balance construction. It was clear that such a balance could cope with practically all the problems likely to face a worker with small amounts of material, and, in general, the further evolution of the microchemical balance has been concerned with modifications of the original Kuhlmann type in order to make weighing operations simpler, more accurate, and less tedious.

Consequently, the torsion type of balance is now only employed for certain limited purposes. Torsion balances, for the rapid "rough" weighing of samples in such fields as organic synthetic work, are still very useful. But for high precision work the use of the beam balance is now almost universal.

To all intents and purposes, then, the microchemical balance in general use to-day is similar in construction to the ordinary analytical balance.

The Kuhlmann type, which for a number of years was unique, has a beam about 3 in. long, the beam being divided into a hundred notches for use with the special rider. The balance was modified by the substitution of a telescope instead of a lens for reading the pointer scale. This enables the worker to sit further from the balance when weighing, so that body heat does not affect the accuracy of the weighing to the same extent.

One of the notable points about the Kuhlmann balance is that since the sensitivity of the balance is exactly 0·001 mg., one scale unit on the pointer scale represents 0·001 mg., at any load up to the maximum, and direct determination of the weight may be made. After such a balance has been in use for some time, arising from wear on the knife-edges, this may well not be true, so that the sensitivity must be determined, and all readings appropriately corrected. Certain American microchemical balances also, must first have the sensitivity determined at various loads, and corrections are then applied to the pointer-scale readings. This usually results from the fact that the three knife-edges are not co-planar. In passing, it may be pointed out that the sensitivity of a new balance may not be exactly 0·001 mg., simply arising from the fact that the sensitivity nut is not precisely at the required point. In such a case it is usually better to correct pointer-scale readings rather than to attempt to adjust the sensitivity nut. However, the other type of fault may be deduced by the variation in sensitivity at different loads.

The Ideal Balance

Consideration of the effect of placing the knife-edges at different levels leads us directly to consideration of the desiderata for a microchemical balance. To ensure a constant sensitivity from zero to full load, the knife-edges, as well as being parallel, should, as has been noted, preferably lie in the same horizontal plane. It is clear that if the beam shows any tendency to bend under load, knife-edges which were originally co-planar will no longer be so. Metal fatigue will produce the same effect. Therefore, the beam should be completely rigid up to the maximum load, which is usually taken to be 20 g. Consequently, the tendency has been, for a long time, to retain the 3-in. beam first developed by Kuhlmann. The production of new alloys now permits of an increase in the length of the beam to 5-in. In such a balance possessing this longer beam there is not the same sensitiveness to outside influences. A balance with the longer beam can therefore be used after a shorter interval of time from assembly or cleaning than is possible with the 3-in. beam, which usually requires several days to come back to normal.

Secondly, it is clear that the effective arms of the balance, that is, the distances from the outer knife-edges to the central one, must be equal. The knife-edges themselves, and particularly the central one, must be ground with the greatest attention to mechanical perfection. Several substances other than agate have been suggested for use as material for knife-edges, of such hardness that the shape will be retained after long use. However, none of these which is sufficiently workable has received sufficient testing to be able to comment on its efficiency.

The position of the rider in relation to the knife-edges requires some consideration. In the Kuhlmann balance the rider notches are cut on the beam itself. These must be cut with the highest precision. On the Kuhlmann type balance, the distance of the rider from the central knife-edge must not vary by more than 7μ on removal and replacement, if a precision of $\pm$ 0·001 mg. is to be maintained. Likewise, its angle of rest must not vary by more than one-fifth of a degree. As a

consequence, the beam must, in the first place, be notched with an accuracy of 7μ, if equivalent movements of the rider on different parts of the beam are to have the same value. But in addition, it is essential that the rider shall always fall into the same position, to ensure satisfactory reproducibility of weighing. Therefore, the notches in the beam should be so cut as to produce this result. Each notch should be in the shape of a perfect and rather narrow V, which will throw the rider to the bottom in a constant position. Many early microchemical balances suffered because of the difficulty of solving the mechanical problem of machining such a beam. It is, however, probably true to say that nowadays a balance by a reputable maker will be unobjectionable in this respect.

Other later types of balance carry the rider on a separate bar co-planar with the knife-edges, which is theoretically the best position. The notches are now cut on this separate bar and not on the beam, presenting an easier task from the point of view of the manufacturer, and therefore likely to be more satisfactory in result. The only comment on this arrangement is that great care must be taken that the position of this rider bar does not get out of adjustment in relation to the beam.

The Nature of the Rider

The rider itself has been of many shapes and of various materials. The Pregl-type rider, of aluminium or of gold wire, is, although not theoretically perfect, satisfactory and convenient in use. Aluminium is the preferred metal, and the weight of the rider should be as accurately as possible 5 mg.

Theoretically, a stick or ring rider has the best shape. A balance with such a stick rider, made of quartz, was developed shortly before the war.[5] However, it was found that the rider lacked the ease of handling of the more usual wire rider, and considerable difficulty was experienced in manipulation, arising largely from changes in alignment of the rider bar (balance beam) and the rider carrier. Further development followed, with more rigid construction, largely through the use of a metal balance case. It is claimed that the new model is very satisfactory, though further information on its performance would be valuable.

The balance case of the microchemical balance has received attention from other points of view, primarily for ease of dismantling, in order to clean the balance. A detachable balance case is now quite a common feature, and has many advantages. Within the case, the balance may be supplied with glass screens, suitably perforated to allow passage of the pan bows, thus cutting down trouble from convection currents.

Reading the Scale

For many workers, one of the greatest sources of difficulty in the use of the microchemical balance lies in the eyestrain arising from the reading of the fine pointer scale. This problem is a serious one, since, as has already been pointed out, the tendency, particularly if the worker is short-sighted or astigmatic, is to approach too closely to the balance case, thus setting up heating disturbances.

A first step towards a solution of this problem was, as we have noted above, the fitting of a telescopic reading to the pointer scale. Along another line, came the application of projected scales to the microchemical balance. Much controversy has raged over the relative merits of these two methods of solving the problem. Doubt has been expressed as to the advisability of applying a strong light beam in the neighbourhood of a balance known to be extremely sensitive to heating effects. Therefore, the projected scale tended to be decried to some extent for a time. The position now may be summed up by saying that for the highest accuracy telescopic reading is probably still to be preferred, although this still leaves partly unsolved the problem of fatigue of the worker. The projected scale balance is equally important, and, with a scale reading to 0·002 mg. (the last place being estimated to 0·001 mg.) has, by the ease of reading, eliminated the strain, and is of full practical value for all general microchemical work.

It is of interest to note that the older type of balance may, with some little ingenuity, be converted to a projected scale balance by some device such as that described by Tuttle and Brown.[6] These authors, using a mirror arrangement, throw an enlarged image of the scale and pointer on to the bench immediately in front of the balance. The image, having a magnification of about eight times, is bright enough to be observed in an ordinarily lighted room.

Aperiodic damped balances were also available from the Continent some years before the war. Air damping is the most usual device employed. It was hoped that by reading a stationary point instead of a moving one, greater accuracy might be attained. However, in practice, this is not found to be so, and the principal advantage to be gained from such balances is some gain in actual speed of weighing, since it is possible to deduce, when no further change in pointer reading occurs, at what point the article being weighed is completely acclimatised. As a rule, however, the damping mechanism requires special handling, and therefore makes the operation of the balance more complex.

The Semi-micro Balance

In this consideration of factors governing the choice of a microchemical balance, it is logical to discuss balances which deal with amounts intermediate between several milligrams and the amounts of ordinary chemical analysis. Early work in this range made use of an ordinary analytical balance. As normally used, such a balance is read to 0·1 mg. Therefore, on a sample of 20 mg., which may be taken as representative of this range, a possible weighing error of 0·5% was immediately introduced.

Balances specifically developed for this range are now available. A representative semi-micro balance is capable of carrying a load of 100 g., and has a sensitivity of 0·01 mg. A balance for this range may be either pointer reading, or may have the advantage of a projected scale. Other refinements of the ordinary microchemical balance, such as an inner glass screen to cut down convection currents, may also be fitted.

The sensitivity of 0·01 mg., for sample weights of 20 mg., gives a considerable margin, since the limit of accuracy of any analysis may be held to lie in the range of 1 in 500 to 1 in 1,000. As is now fairly generally known, the true sensitivity of a good analytical balance should be as low as 0·05 mg., and probably lies considerably lower than this figure. From this it can be deduced that if a weighing procedure which takes full

5 Kuck and Loewenstein, *J. Chem. Educ.*, 1940, **17**, 171.

6 Tuttle and Brown, *Ind. Eng. Chem., Anal. Ed*, 1944, **16**, 645.

advantage of the sensitivity of the balance is employed, the weighing error will probably lie inside the range of accuracy of the determination.

Using the method of swings[7] which is essentially the method employed in the Kuhlmann type of balance, and determining the sensitivity of the balance over the load range anticipated, an analytical balance with an average accuracy is quite competent to deal with all normal semi-microanalyses. Even, indeed, if only an analytical balance which has undergone quite intensive usage is available, it is often possible, if the knife-edges have not been actually damaged, to meet all the requirements of the semi-micro-analyst by careful cleaning and adjustment, and subsequent retention of the balance for semi-micro work only.

With such a balance so used, the sensitivity is determined at zero load, and for loads of 1, 2, 5, 10, 20 and 50 g. A graph is drawn of sensitivity against load, thus enabling the precise weight of any load over this range to be calculated. At the same time the precision of the balance is determined. It may be assumed that an analytical balance with a precision of $\pm$ 0·05 mg. or better is quite suitable for use as a semi-micro balance. Thus, a typical balance had a sensitivity (at zero load) of 0·019 mg., and the average deviation of a single weighing was $\pm$ 1·6 pointer scale units (each scale division being read as ten units). This gives a precision of $\pm$ 1·6 $\times$ 0·019 mg., or 0·030 mg., so that such a balance is eminently suited to small-scale practical work.

The Analytical Balance for Micro Work

Because of success in applying the analytical balance to semi-micro work, several authors have investigated the possibility of using the ordinary analytical balance for even smaller samples.[8] A 5-mg. rider balance, chosen from among student balances, was taken for some of this work. It was found to have a sensitivity of 0·022 mg., and a precision for duplicate weighings of $\pm$0·022 mg. In organic analyses, using samples of about 10 mg., quite accurate results were obtained. Thus, for carbon and hydrogen determinations, an accuracy of $\pm$ 0·4% was achieved. Belcher and Godbert[9] demand an accuracy of $\pm$ 0·2 for semi-micro determinations of this type, and elsewhere,[10] for normal micro-analysis, an accuracy of $\pm$ 0·2–0·3% is quoted as reasonable.

Similar procedures were also devised for inorganic analyses on the same scale, with comparable results. However, it is safe to say that such analyses could not be recommended as a general practice. They should, however, be borne in mind for occasional application where a sample available might be too small for semi-microanalysis, while a microchemical balance was not available.

Finally, in all cases considered, whether the balance is a full microchemical one, a semi-micro one, or an analytical balance specially reserved for small-scale work, whether it is pointer reading or projected-scale reading, it must be emphasised that its mechanical parts must function properly. In particular, the releasing and arresting mechanism should be properly adjusted and smooth-working, so that on release there will be no jolting of the mechanism, and that the pointer will automatically give a moderate swing, of the order of five scale divisions, on the pointer scale. Similarly, on arrest there should be no opportunity for damage to the knife-edges or other working parts.

7 Belcher and Godbert, *Semi-micro Quantitative Organic Analysis*, Longmans, 1945, p. 10 ff.

8 Niederl *et al.*, *Ind. Eng. Chem., Anal. Ed.*, 1939, **11**, 412; Benedetti-Pichler and Paulson, *Mik.*, 1939, **27**, 339.

9 Belcher and Godbert *loc. cit.*, p. 6.

10 Pregl-Grant, *Quantitative Organic Microanalysis*, Fourth English Edition, Churchill, 1945, p. 33.

The Intermittent A.C. Arc in Spectrography

IT has already been noted in these columns[1] that for accuracy and reproducibility in quantitative spectrographic analysis the intermittent A.C. arc as source is to be preferred to the D.C. arc. A circuit diagram is given for such an arc in a recent paper.[2] The electrodes are of graphite, and are drilled at one end on a lathe to form a roughly hemispherical cup. This is polished, while still in the lathe, with a piece of stiff paper, so that liquid will not penetrate into the graphite. A uniform drop of solution, containing the unknown and an internal standard, is then applied to each cup, and rapidly evaporated so as to produce a thin, smooth and firmly adhering crust of solid. For the determination of lead, tin and chromium, bismuth was used as internal standard, the recommended pairs of lines being :—Pb 2833·1 A, Bi 2898·0 A; Sn 2863·3 A, Bi 2898·0 A; Cr 2835·6 A, Bi 2898·0 A. A medium quartz spectrograph was sufficient for all analyses except where large amounts of iron were present. In such cases iron was removed, or a large quartz spectrograph was necessary.

Iron has been used itself as an internal standard for a wide range of elements, comprising :—Mg, Mn, Pb, Sn, Cr, Al, V, Mo, Cd, Cu, Zn, Ni, Sr, Co. The method has been found unsuitable for Ba, Ca, W, Ag, Ti, Si, and, of course iron itself.

For this range the large quartz spectrograph is necessary. Six iron lines serve for the determination of the various elements, as follows :—Fe 2802·5 A for Mg (less than 0·2%, 2802·7 A ; more than 0·1%, 2779·9 A) and Mn (2798·3 A). Fe 2845·6 A for Pb (2833·1 A), Sn (2840·0 A) and Cr (2849·8 A). Fe 3091·6 A for Al (3082·2 A), V (more than 0·25%, 3110·7 A ; less than 0·5%, 3185·4 A), and Mo (3158·2 A). Fe 3254·4 A for Cd (3261·1 A). Fe 3286·8 A for Cu (3274·0 A). Fe 3450·3 A for Zn (3345·0 A), Co (3405·1 A), Ni (3461·7 A) and Sr (3464·5 A).

Specimen results, reproducibility and accuracy are fully discussed. In general, an accuracy of $\pm$ 5–10% of the proportion of the element present is achieved.

1 *Metallurgia*, 1946, **34**, 165.

2 McClelland, *Analyst*, 1946, **71**, 130.

Erratum

In the discussion on the influence of raw materials on the property of whiteheart malleable cast iron, on page 125 in July issue, Mr. Makemson is reported to have said : "production for two years was 200,000 tons a week." This is an obvious error ; it should have read 3,000 tons a week.

Nickel - Antimony - Lead - Copper Bearing Alloys

By J. T. Eash

AMONG the commonly used bronzes for heavy-duty bearings the 10% tin, 10% lead-copper alloy is most commonly used. To conserve tin an investigation has been carried out to develop an alloy of low tin content that could be used for bearings and having metallurgical properties similar to this 80-10-10 bronze. In carrying out the tests, such properties as microstructure, hardness, compressive yield strength, ductility and castability have been considered, and certain simple wear tests have been made.

In a leaded bronze used as a bearing metal there must be a uniform distribution of lead, and previous investigations have shown that, in general, the addition of nickel to lead-copper alloys has a favourable effect on lead distribution. It was found that while the agglomeration of lead was started in copper-lead alloys by the addition of nickel, it was incomplete, and, in addition, the hardness of the nickel-lead-copper alloys was about 35 Brinell as compared with 65 Brinell for tin bronze. Further improvement was therefore required, and consequently the effects of other alloy additions to the nickel-lead-copper base were investigated. The effects of various additional alloys on the hardness, fracture and microstructure are summarised in Table I. From all these results, antimony appeared the most promising in dispersing the lead and 1% was quite effective. The hardness of these antimony alloys was about 50 Brinell which was appreciably above that of the straight nickel-lead-copper alloys, while the fractures of the bushing containing 2·5 to 8% nickel and 1% antimony were sound and the most satisfactory of those produced.

A series of nickel-antimony-lead-copper alloys was prepared and cast into bushings for measuring castability, discs for wear tests and cast-to-shape tensile bars. Compression specimens 0·505 in. dia. by 1 in. long were machined from the tensile bars, and samples for microexamination were cut from the wear discs, and examined close to the wearing surface, since it is possible for the structure of leaded alloys to vary in different parts of a casting. The results obtained from these various alloys are given in Table II.

Considering in detail the wear data, it is noted that the 10% lead-copper alloy with 1% antimony or 2·5% nickel alone was inferior to the 80-10-10 alloy. Not until 2·2 to 10% nickel and 1% antimony were added together did the wear improve substantially; with 2·2 to 8% nickel, wear resistance superior to 80-10-10 was obtained and at the 10% nickel level the wear was equal. Raising the antimony to 1·4% caused an increase in wear loss. The wear of the mating steel disc in the Amsler wear test in every case was extremely low and the torque

TABLE I.—Effect of Alloy Additions to 10% Lead-Copper Alloy.

Composition %			Brinell Hardness	Lead in Microstructure	Fracture of Bushing
Pb	Ni	Other			
10	—	0·5 Zr	40	Some agglomerate lead; some grain boundary lead.	Considerable shrinkage.
10	—	0·5 Ca, 0·5 Li	—	Porous.	Shrinkage and gas porosity.
10	—	0·4 S, 0·5 Zr	52	Intergranular lead and sulphide; massive particles.	Shrinkage and gas porosity.
30	2	5 Mn	42	Massive intergranular lead.	Much dross.
10	5	1 Mn	52	Few lead particles.	Lead sweat.
10	5	0·5 Mn, 1 Sb	50	Uniform distribution of agglomerate lead.	Heavy lead sweat.
10	5	1 As	50	Lead films and agglomerate lead.	Lead sweat—slight shrinkage.
10	5	1 Bi	40	Lead films.	Lead sweat—slight shrinkage.
10	5	1 Se	45	Massive intergranular lead and selenide.	Slight lead sweat and shrinkage.
10	2·5	1 Sb	45	Uniform distribution of agglomerate lead.	Good.
10	5	1 Sb	51	Uniform distribution of agglomerate lead.	Good.
10	8	1 Sb	50	Uniform distribution of small lead particles.	Good.

TABLE II.—Properties of Bearing Alloys.

Composition %				Wear Test 240,000 Revs. Wt. Loss in Mg	Brinell Hardness	Tensile Properties			Compression Yield 0·5% Compression Tons/sq. in.
Pb	Ni	Sb	Sn			Ultimate Stress Tons/sq. in.	Yield Stress 0·5 Extension Tons/sq. in.	Elongation %	
9·81	—	—	(10)*	180	67	16·2	7·9	18	8·8
(10)	(2·5)	—	—	314	34	4·4	2·0	5	—
(10)	—	(1)	—	481	45	6·3	3·6	5	—
10·6	2·2	0·96	—	95	46	5·1	4·1	3	—
9·0	8·0	0·96	—	50	53	9·0	5·9	6	6·9
(10)	(10)	(1)	—	188	52	9·5	5·1	9	—
9·6	8·1	1·4	—	372	60	11·2	5·8	9	—
8·3	7·7	0·99	1·09	125	63	10·6	6·6	6	8·2
10·9	7·7	0·85	2·86	43	71	12·7	9·5	4	10·2
10·3	7·3	0·88	5·00	70	87	15·6	12·6	2	12·9

From *Metals Technology*, 1945, Vol. 12, No. 8, and A.I.M.M.E. Technical Publication, 1937, pp. 1–11.

developed was also very low, being only 0·9–1·4 kg. cm.

From the standpoint of wear resistance alone, the bearing containing 2·2% nickel, 1% antimony, 10% lead would be superior to the 80-10-10 alloy. Mechanical properties, however, lead to the selection of a higher nickel content, and with 8% nickel, 1% antimony and 10% lead values were obtained that were about 70% those obtained with the 88-10-10 alloy. Increasing the nickel and antimony still further was effective in raising the tensile strength, but was detrimental to the wear resistance. The tensile and yield strengths of the 8% nickel, 1% antimony and 10% lead alloy were found to increase directly with tin additions up to 5% and to maintain excellent wearing properties. Ductility, however, became lower and the yield in compression increased with the tin content. It was concluded that in order to match the yield strength of the 80-10-10 alloy in either tension or compression 2% of tin must be added to the 8% nickel, 1% antimony and 10% lead alloy.

In the 10% lead, 2·5% nickel-copper alloy, the intergranular formation of lead was partially disrupted, and with 1% antimony, the dispersion of the lead proceeded still further. When the two metals were added in combination further change occurred, the lead being more finely dispersed, and a new phase appeared which increased with the nickel content. The addition of tin resulted in the formation of a finely dispersed constituent which was located in interdendritic areas of high tin content.

Experiments on the effect of melting temperature of the 8% nickel, 1% antimony, 10% lead alloy, showed that superheating should be avoided as it promoted the formation of intergranular lead films. Heating temperature during melting should be limited to 1,230° C. and the pouring temperature determined by the section being cast. To determine the effect of section size, castings of 2½ in. dia. were made having thicknesses of ½, 1 and 2 in., and a ½-in. thick disc having a chill around the periphery was also cast from the same heat. The chill cast specimen had a high wear rate while all the sand-cast specimens had low wear rates. The 1-in. section was equal to the 80-10-10 alloy and the other two were much superior. The nickel-antimony-lead-copper alloy also had good shrinkage characteristics, and sound castings could be readily produced in ordinary green sand, if a cement-bonded core was used.

Materials of Construction in the Petroleum Industry

By T. McLean Jasper

THE use of materials in this industry calls for considerable knowledge of their characteristics so that the principles of economic engineering may be satisfied in their use. Economic engineering involves the selection of materials so that their application will produce the resultant structure for the least total cost in the service to be undertaken, considering the safety of operations and the life of the plant.

The producer of oil wells for the petroleum industry will in the future be required to drill to greater depths. This requires qualities in the engineering materials of increased strength and resistance to abrasion, fatigue, and corrosion. Not all of these qualities need be combined for all of the engineering materials used in production. It does, however, imply that good technical knowledge should be applied in the proper selection and application of such materials.

From *Mech. Eng.*, **68**, 1946, pp. 423–4.

The alloying elements in steel that will increase strength most economically are manganese and carbon. In casing, where it is necessary to increase resistance against collapse, the least expensive method is cold compression of the tubes in the transverse direction. Whatever may increase the compressive yield strength of steel will also perform the same function for thick pipe. For resistance to abrasion, the most important alloying materials for drilling operations are manganese, molybdenum and tungsten. A proper combination with carbon is important in this connection also. Abrasion resistance requires a combination of properties involving toughness, resilience, surface hardness for some problems, or a combination of one or more of these properties in order to produce the best results.

To increase the endurance limit or resistance to fatigue, nearly any method which increases the ultimate strength of steel will perform this function. For resistance to corrosion in steel at production temperatures, chromium and/or nickel are the most direct and successful alloying elements for the type of service associated with the production of petroleum from the ground. To perform the desired functions most economically, requires experience as to which one or more, if any, of the alloying materials should be used and how much.

Crude oils are transported in tankers, inland barges, pipe lines, and tank cars. Gases, except those which can most easily be liquefied, are transported in pipe lines. These oils are not highly corrosive in general and therefore plain steel containers are the rule. Where corrosion becomes a problem in connection with the transportation of crude products it is generally from the outside of the container and results from weathering or soil contact. To resist this type of corrosion steel is one of the most expensive materials. Where the corrosion problem becomes important, as in particular types of soil corrosion, a coating of tar or asphalt wrapped in fabric is used for pipe coating and becomes much more economical than alloying or thickening the steel. Recent developments indicate that some of the plastic coatings may fit into this situation as they become better understood and less expensive. Cathodic protection is also used in some localities.

Transportation of the by-products of refining can sometimes result in exceedingly difficult problems of corrosion. The ordinary steel tank car, pipe line, tanker, inland barge, or storage tank is sufficient for many of the common motor fuels when unprotected internally. As the synthetic products of refining become increasingly available, the question of combating corrosion of the container and the deterioration of the product becomes more important. Lubricating oil and high-octane gasoline should be absolutely free from fine particles of iron oxide.

To an even greater extent the refinery is becoming a chemical plant with all of the problems of such a plant. Solvents, as well as other products used for plastics which must have no iron contaminations, require in some instances that we use aluminium for the tank which is built into the tank car and the storage facility.

The application of materials to the problems of refining calls for a greater knowledge of materials selection than

that associated with production or transportation. Refining involves high-temperature and high-pressure operations. Many refining processes are also associated with low-temperature operations, and these tend to reduce the working pressures at which the low temperatures are effective. In conjunction with both the high-temperature and low-temperature operations, conditions may exist of differential cyclic stresses which become of considerable importance when the temperature changes are rapid. Then the conditions of operation involve heavy sections of material. Little is known about fatigue at high or low temperatures except that, at high temperatures the values are reduced, and at low temperatures the values are increased but the materials have a greater tendency toward brittleness.

Chemical activity increases rapidly as temperatures of operation are increased. On the other hand, strength of materials decreases rapidly at the higher temperatures of refining. Metal temperatures of 1,200°F, and a container pressure of 3,000 psi are encountered in oil-refining processes. Ceramic-material temperatures may reach much higher values than that mentioned. The desired chemical reactions, however, are more rapid and more complete at the higher temperatures of operation; so much so that economic engineering sometimes calls for very expensive materials because of their greatly enhanced resistance to the conditions of refining.

The alloying materials most suited to improving the strength of steel at elevated temperatures are chromium, molybdenum, and nickel. Often associated with these are vanadium, columbium, or titanium. For pressure-vessel steel, tungsten is not used because of its cost and the trouble encountered in forming steel containing it. However, tungsten is used with nickel and molybdenum, or chromium with nickel may also be used in high-strength bolts for high-temperature service.

The alloying materials most suited to improve the ductility and shock resistance of steel at low temperatures are nickel, manganese and silicon. There is no doubt but that a clean well-deoxidised steel gives the best results. For intermediate low temperatures, manganese and silicon, properly balanced, can meet the general specifications called for.

The alloying materials most suited to enhance the corrosion resistance to high-temperature refinery operations are chromium and nickel. Along with these, small amounts of titanium or columbium are used in many instances to render the steel more satisfactory.

Since the most economical steel for load carrying is not necessarily the same as that required for corrosion resistance, the economic engineering point of view calls, in many instances, for an inner lining of more expensive corrosion-resistant material and an outer and thicker layer of higher-strength load-carrying material. On the other hand, for thin-walled vessels where corrosion resistance is of importance, a solid corrosion-resistant metal is the most economical. As the pressure of operation increases, the thickness increases and a dual metal offers the most appropriate selection. Clad materials (the so-called ply metals), spot-welded liners, plug-welded liners, and inner layers of corrosion-resistant metals are all used.

A considerable amount of ceramic materials of various kinds are used in the refining operations. Such materials are used on the inside of vessels in some processes to insulate the outer steel from the high temperatures of the operation; and sometimes to resist the corrosive attack of the petroleum materials being processed. In this manner, a reduction in steel wall thickness can be made. Such liners, of course, also have the effect of reducing heat losses to the outside of vessels.

For many of the functions in a refinery, the use of materials follows the demands for transportation and storage. The operation of refinery equipment sometimes involves large temperature differential stresses. In many cases these stresses have not been considered in the designs. It should be pointed out that temperature stresses due to rapid cooling are sometimes responsible for operating failures of important structures. These failures can be reduced, and the hazard of failure practically eliminated, if attention is paid to cooling rates, and to a reduction of the thickness of the materials to be cooled.

The author's experience indicates that a vessel 4 in. thick is at least six times as hazardous as a vessel 2 in. thick when too rapid cooling is practiced, provided both vessels are designed with the same operating factor of safety. In one instance the operating temperature of certain vessels was 900°F. Quick cooling of such vessels, in bringing them out of service for cleaning out the coke, was responsible for stress differentials which could, and did, cause vessel failure. This fact led to the selection of steel with a greater long-time strength at 900°F, resulting in reduction of the needed thickness from 4 in. to 2 in. for the same vessel size. It should be apparent, therefore, that there are factors in the use of materials where, in many cases the increase of plate thickness decreases the safety of such equipment.

The general problem of materials in the petroleum industry applies with very little difference to many other industries. The solution of numerous difficulties is brought about by proceeding from a knowledge of the requirements of a new process and the experience in the application of materials to known conditions of service in other similar processes. There is a certain amount of experiment associated with producing any new advancement but if simple tests are made to simulate the conditions of the anticipated service, it is usually not long before a solution unfolds itself. If the new process is economically right it then becomes important to bring the equipment to the most appropriate degree of efficient application of materials.

Acidic Atmosphere Evaluation of Cleaning on the Corrosion of Steel

By Chester W. Smith

CLEANING methods used on steel produce differences in the corrodibility of the surface with exposure to the normal elements present indoors in industrial atmospheres. The "acidic atmosphere cell" which was devised, produced a type of corrosion similar to that encountered in actual exposure. This simple accelerated corrosion test incorporated many of the significant factors present in regularly encountered shop atmospheres—*e.g.*, moisture, acidic gases, oxygen, temperature variations. This cell was used as a means of evaluating different cleaning materials and methods in regard to their effect upon the corrodibility of bright steel. Highly finished steel cleaned in regular production is allowed many times to stand for periods or stored without the protection of an oil film. The effect of cleaning upon the subsequent corrodibility of the steel was the primary consideration of the present study. Cleaning

From *Electrochem. Soc.*, Preprint, 89–25, April, 1946.

methods using either abrasives, or alkaline compounds, or volatile or partially volatile solvents, and emulsified solvents were compared as to effects on bright steel and when exposed in the "acidic atmosphere cell." Emulsified solvent cleaning materials which leave a film of non-volatile emulsifier render steel surfaces the least corrodible. Steel cleaned with such materials can usually be further processed by inspection, machining, bonderising or painting without any additional cleaning step and may be held over for indefinite periods without corroding in an industrial atmosphere.

A Test for Hydrogen Embrittlement and its Application to 17% Chromium Stainless Steel Wire

By C. A. Zapffe and M. Eleanor Haslem

TENSILE tests, impact tests and bend tests are commonly used for detecting hydrogen embrittlement. Bend tests have usually been of three general types: (1) Fatigue; (2) reversed bend; and (3) single bend. The first two types, although widely used, are inaccurate because their measurements are counteracted by an ageing effect, and the third type has not been developed to its fullest extent. An investigation was, therefore, carried out to develop a method for measuring hydrogen embrittlement that avoids certain errors complicating previously used methods, to explore in a preliminary way some of the fundamental factors controlling embrittlement, and to apply the measurements specifically to stainless grades of steel, whose sensitivity or insensitivity to hydrogen embrittlement has never been clearly defined.

A simple apparatus, Fig. 1, was constructed using a synchronised motor A with a gear-reduction ratio 600–1. A card, fixed at one end to a wooden pully B, on the drive shaft of the motor follows the periphery of the horizontal semicircular base C upon which angular marking from 0°–180° are indicated. A hook is attached to the other end of this cord, which catches the travelling arm D in the 0° position, and this travelling arm is drilled longitudinally to receive one end of the wire specimen, the hole being drilled tangent to the axial pin E, which has a radius of $\frac{1}{16}$ in. A fixed arm F backs up the wire and stabilises the specimen without allowing tensile or torsion stresses to develop. Exactly 30 secs. after each experiment was completed the test specimen was placed in this machine and the angle of bend measured.

Experiments were made to determine the hydrogen embrittlement resulting from: (1) Cathodic charging using 10% sodium hydroxide and 10% sulphuric acid electrolytes; and (2) acid pickling with different pickling reagents. As a result of preliminary tests and because such a steel was known to show sensitivity to hydrogen during processing, a 17% chromium, 1% carbon steel was used as the test material in the form of 0·062 wire having a tensile strength of 50 tons/sq. in.

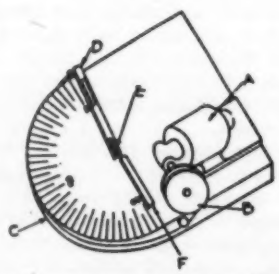

Fig. 1.—Apparatus for measuring hydrogen embrittlement in wire by means of a steadily applied single bend.

As a result of the various tests, it was found that with increasing pickling time, embrittlement increased along a curve having three stages. The first stage was an incubation period in which atomic hydrogen was absorbed into the lattice without decreasing the bend value. Simultaneously hydrogen was precipitated from the lattice into crystallographic lattice voids where it gathered under constantly increasing pressure. In the second stage a sudden decline in bend value occurred which was considered to result from the precipitating gas, suddenly attaining the critical pressure necessary to cause embrittlement, while in third and last stage a gradual slope was obtained in the curve which plotted logarithmically as a straight line and was considered to represent stabilised inward diffusion once the critical precipitation pressure was reached and maintained near the surface.

With increasing temperature during charging, embrittlement increased when all other factors were constant. This effect was especially prominent in short pickling periods which occurred in the first stage of the curve at low temperature and therefore caused no embrittlement, but which exceeded the first stage of the curve at higher temperatures and therefore led to embrittlement. Embrittlement was not directly proportional to current density, a maximum embrittling effect occurred close to 1·0 amp./sq. in. Cathodic charging provided identical results whether the electrolyte was 10% sodium hydroxide or 10% sulphuric acid.

With 17% chromium, 1% carbon stainless steel, quinoline ethiodide and arsenic, although true inhibitors of hydrogen embrittlement, when added in certain concentrations in straight acid pickling, both have a range of concentration in which embrittlement was badly aggravated. Arsenic also showed no effect at all in cathodic pickling. Sulphide iron, supplied by dissolving hydrogen sulphide in the solution caused a marked lowering of the bend value, consequently contamination of the bath from pickling free-machining grades of steel should be watched. In ageing, the bend value first decreased, which was ascribed to hydrogen atoms continuing to precipitate from the lattice into the lattice voids after charging had stopped. This phenomenon of an ageing minimum was so marked that specimens charged in the first stage and showing a perfect bend might subsequently spontaneously embrittle themselves. Hydrogen embrittlement was also found to confer no detected changes on hardness or electrical resistivity.

In general the results of the investigation showed strong evidence in favour of the planar-pressure theory for hydrogen embrittlement and were definitely inconsistent with theories based upon lattice distortion or compound formation.

From *Metals Technology*, 1946, Vol. 13, and A.I.M.M.E. Technical Publication No. 1954, pp. 1–28.

STEELS IN ENEMY AIRCRAFT.

SOME copies of the above book are still available. Order direct from the Kennedy Press, Ltd. 31 King Street West, Manchester, 3, enclosing 10/6.

Heat-Treating Beryllium Copper

By H. G. Williams

BERYLLIUM copper offers the user of springs and stampings a unique combination of properties. A tensile strength of nearly 90 tons/sq. in. places beryllium copper in the steel class, yet it is corrosion resistant, non-magnetic, and conducts electricity four to five times as well as steel. It has higher endurance strength, greater conductivity, and can be used at igherh temperatures than any copper alloy.

Its exceptional properties are obtained largely as a result of heat treatment. It can be formed into intricate shapes while soft and ductile and then hardening to spring temper with a low temperature heat treatment. The same hardening heat-treatment also can be used to free the structure of internal stresses, resulting in greater stability than found in spring alloys that gain their temper from cold work.

If only one characteristic is involved, the selection of the proper heat treatment is relatively easy. More often several properties are wanted, some two of which may not ordinarily develop to the greatest degree at a single heat treatment. The proper alloy composition, the amount of cold work prior to heat treatment, the solution anneal before the cold work, and the temperature used for the heat treatment are all factors bearing on the relative rate at which the various properties develop. To obtain full advantage of what beryllium copper offers requires complete knowledge of the effects of these factors.

The beryllium-copper alloy most commonly used for spring applications includes a composition range of 1·90 to 2·20% beryllium, 0·5 max. per cent. of nickel or cobalt, 0·5 max. per cent. total impurities,, and balance of copper. This alloy is solution annealed by heating to 760° to 800° C. and then quenching rapidly. Control of the time and temperature of the anneal and rate of the quench affects the grain size and the amount of beryllium available for hardening. Accurate control of the annealing operation is necessary as the solution anneal not only softens the alloy, but puts the beryllium into solution with the copper, making it available for precipitation hardening process which follows. The final properties obtainable depend upon the amount of beryllium in solution and available for hardening.

Hardening heat treatment is usually given after the part is formed and consists of heating between 285° to 400° C. for times ranging from several hours to a few minutes. The mechanism involved is commonly referred to as precipitation hardening, wherein the unstable supersaturated beryllium copper solid solution is decomposed by the heat treatment to form a gamma phase precipitate that increases the slip-resistance of the crystals. The precipitated particles have been considered submicroscopic in size, but recent work with the electron microscope indicates that the hardening develops during atomic rearrangement preparatory to actual precipitation.

A problem in nomenclature is here presented if the hardening is due to an ordering rather than a precipitation, although if the time at heat-treating temperature is long enough, a definite precipitation does occur with formation of discrete gamma phase particles. However, this point is not reached until peak hardness is passed and a softening of the alloy occurs.

At 285° C. it takes five to eight hours to bring half hard beryllium copper to its peak hardness. At 315° C. the time is one and a half to two hours, at 345° C. about twenty minutes, and at 370° C. peak hardness will be reached in about twelve minutes. At 400° C. the time is about five minutes, but the hardness reached is not as high as at the lower temperature.

If the major requirement is high hardness for wear resistance as in bearing applications, or high tensile strength and yield strength and yield point for structural parts such as aircraft door hinges, any of these temperatures can be used with the corresponding heat-treating time. The use of 315° C. with a time of one to two hours requires less critical control of time and temperature and is more practical for the shop not well equipped with control apparatus.

Parts of heavy cross-section are also best hardened at the lower temperatures to gain uniform hardening throughout the piece. This applies to beryllium copper hammers, chisels, and other parts used as sparkless tools. In some forms of heavy cross-section it is desirable to have maximum surface hardness for wear resistance, but brittleness is detrimental. Here a high hardening temperature can be used to give a case-hardening effect with the surface at peak hardness and the core underaged with consequent greater ductility.

In making high-quality springs much advantage can be derived by using carefully controlled higher hardening temperatures. As the heat-treating temperature is increased, the rate of stress relief increases so that at the higher temperatures, around 370° C., the point of minimum drift closely approximates peak tensile strength. Since the same degree of stress relief can be attained at a higher elastic limit, the amount of drift is lower, resulting in a spring of much greater stability.

Annealed stock is usually selected only when intricate forming or drawing is needed. Maximum working stresses are usually in the formed portion of the part, where the material receives considerable cold work. But the unworked flat portion is the only section that offers a chance of testing for hardness. Any heat treatment giving maximum hardness on such a flat section will be sufficiently long to cause overaging of the cold worked portion of the spring because of the effect of cold work on the rate of hardening.

From *The Iron Age*, **156**, pp. 58-64.

Uranium and Radium in the Postwar Period

By S. C. Lind

NEW scientific interest attaches to uranium through the suggestion that it may be a member of a new rare earth series beginning with actinium. Thorium, protactinium, and uranium would then be second, third, and fourth members respectively of the new series. Uranium, therefore, would now be placed below neodymium in the periodic table of elements instead of below tungsten as at present. Uranium having the heaviest natural atom will continue to be the springboard for extension of the series into the transuranial elements. Already we have neptunium and plutonium by neutron reaction on uranium and recently $Seaborg_3$ announced new elements 95 and 96 produced by bombarding uranium and plutonium with forty million volt helium ions.

From *Electrochem. Soc.* Preprint 89-14, April, 1946.

Magnesium Alloy Corrosion

is no longer a critical technical problem

Pit Corrosion of Magnesium Alloy Castings

HATFIELD, ENGLAND

To the Readers of METAL PROGRESS:

I have read with interest of Mr. Miller's experiences with magnesium castings as given on p. 752 of the October 1944 issue of *Metal Progress*. My own experience is that every corrosion problem is one alone; each requires personal investigation on the spot by a competent investigator before intelligent comments can be passed. Atmospheric conditions here at Hatfield are described by us as "rural" since there is virtually no industrial polution. Under these conditions we have had small magnesium-base alloy castings on atmospheric exposure test for 4 years. The nominal composition of these castings is 8.0% aluminum, 0.4% zinc, 0.3% manganese. They are supplied as castings to British specification DTD.59.A, whose impurity limits are rather higher than those stipulated in U. S. Army and Navy specification AN-QQ-M-56.

The castings in question have been continuously exposed to the natural elements. Included in the series is a casting which has been treated by the British Royal Aircraft Establishment "half-hot chromate process" only. As the excellent condition of this casting may be of interest a photograph is reproduced.

As regards storage: It is desirable that such parts be kept in a clean dry place. If storage conditions are not good, then the application of a suitable organic protective may be worth while.

G. W. WEEKS
Aircraft Division Metallurgist
The de Havilland Aircraft Co., Ltd.

On perusal of recent American Technical Press this unsolicited testimonial was discovered.

ELEKTRON
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Silicon-Impregnated Steels

By Harry K. Ihrig.

HIGH silicon iron alloys, whilst very resistant to corrosion, heat, and wear, have been limited in use in that they can be made only in the cast form. They cannot be machined and are very brittle. If the silicon alloy were only in the outer layers of the components and were supported by a soft ductile core, the desirable properties of the silicon alloy would be obtained with the elimination of most of the undesirable ones. This has been made possible by a process of impregnation of steels with silicon.

The impregnation of metals with silicon is not new, but the methods applied until recently were not a commercial proposition. Recently, however, a new technique has been applied on a scale large enough for it to merit commercial consideration. A source of cheap silicon is required and one of the cheapest of these is silicon carbide. Pure silicon carbide contains 70% silicon. Amorphous silicon carbide, a by-product of the manufacture of crystalline silicon carbide abrasives, is a cheap source of silicon carbide. It contains about 80% of the desired compound. Silicon carbide is very inert in air even at very high temperatures, in the presence of chlorine at high temperatures, however, silicon is released for the impregnation of metals and the reaction is the basis of the commerical process described.

For silicon impregnation the rotating retort type furnace, similar to those used for carburising, seems to be the most satisfactory. Provision is made for the introduction of chlorine. The oxidation resistant, high-alloy type retorts do not give as good results as carbon steel retorts. An ordinary 10 in. heavy seamless low-carbon steel tube, with a heated zone about 5 ft. long, has a life of 50 to 60 runs before failing from oxidation on the outside. The retort is arranged to rotate at the rate of about one revolution in two to three minutes. An efficient type of furnace with two bases and retorts is in operation on the process. The heating cover is removed from one retort upon completion of the required time at temperature and transferred to a previously loaded retort on a second base. The first retort then can be unloaded and reloaded while the second is being heated. It is not necessary to cool the furnace to unload a retort.

The metal parts to be treated are charged into the retort, together with silicon carbide, so that there is even distribution, and the furnace heated to about 1,000° C. When the temperature is reached, chlorine is started in at one end for half the run, and switched to the other end. A case of about 0·015 in. is produced in 30 mins. at treating temperature. In about an hour the case will be 0·020 in., in two hours about a 0·035 in. case, and in three hours a case of about 0·050 in. When the parts have been impregnated they may be removed as soon as they are cool enough to handle. They are not quenched or otherwise heat-treated since the silicon alloy case cannot be changed by any known heat treatment. The parts come out fairly clean and may be brushed if necessary.

The most corrosion and wear-resistant cases are formed on low-carbon, low-sulphur steels. The carbon should be below 0·25% and the sulphur below 0·04%. Most alloying elements adversely affect the properties of the silicon case. Cast or wrought steels may be used. White and malleable irons have been treated successfully. Ordinary grey cast irons tend to swell badly at treating temperatures and the cases on them are relatively soft and porous.

Sections under $\frac{1}{8}$ in. in thickness are likely to bend in the rotating retort at the treating temperature. Very thin sections tend to be treated all the way through and hence are brittle. Sharp corners should be avoided.

When parts are to be held in close tolerances, or where smooth surfaces are required, they should be rough machined or otherwise fabricated with an allowance of 0·010 in. extra stock on a side which is removed by grinding after the silicon impregnation.

Threaded parts either must be threaded after treatment by removing the case in the threaded area or should be threaded undersize to allow for a slight swell. This varies with different sized threads and sections so trial parts should be run to determine this. After it is determined, it remains constant from run to run.

From *The Iron Age*, April 4, 1946.

Physical Properties of Copper-Manganese-Zinc Alloys

By R. S. Dean, J. R. Long, T. R. Graham and R. G. Feustel

IN the course of an investigation on establishing the limits of the alpha solid solution range of the copper-manganese-zinc alloy system, enough of certain of the alloys were prepared to permit determination of their physical properties. The 60% copper alloys at present investigated were part of that series, and crossed through the alpha field into the adjacent alpha plus beta and alpha plus X fields. The chemical compositions of the alloys tested are given in Table I. They were finished into sheet by cold-rolling and intermediate annealing procedures calculated to produce sheet $\frac{1}{16}$ in. thick, cold-reduced by 20, 40, 60 and 80% in thickness.

Physical properties were determined on standard flat tensile specimens with 4 in. gauge length. The properties were obtained on cold-worked material and on material annealed at temperatures ranging from 350° to 815° C.

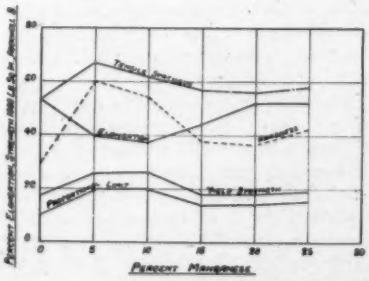

Fig. 1.—Physical properties of copper manganese-zinc alloys containing 60% copper, 0 to 25% manganese, after annealing at 650° C.

after 60% cold reduction. Since an annealing temperature of 650° C. was used for all intermediate annealing in fabrication of the sheet, the material representative of the zero per cent. cold work state was also annealed at this temperature. The physical properties of the alloy after annealing at 650° C. are given in Fig. I.

From *Metals Technology*, 1945, Vol. 13, No. 1, and A.I.M.M.E. Technical Publication, No. 1956, pp. 1–12).

TABLE I.—COMPOSITION OF ALLOYS INVESTIGATED.

Alloy	Copper	Manganese	Zinc	Aluminium	Iron	Silicon
17	60·1	5·0	35·1	0·04	0·008	Nil
18	60·5	9·8	29·8	0·02	0·005	Nil
19	60·5	14·6	25·0	0·02	0·005	Nil
20	60·2	19·5	20·5	0·02	0·005	Nil
21	59·7	24·8	15·0	0·02	0·005	Nil
11936	60·6	28·8	10·7	—	—	—
10934	59·3	35·1	5·4	0·05	0·02	Nil

The results obtained showed that in copper-manganese-zinc alloys containing 60% copper and 5 to 25% manganese, annealed at 650° C. the tensile strength varied from 30·0 to 24·5 tons per sq. in. The maximum strength occurred in the 5% manganese alloy, which had a tensile strength of 30 tons per sq. in. (67,000 lb. per sq. in.), a yield strength of 12·5 tons per sq. in. (27,500 lb. per sq. in), an elongation of 39% and a Rockwell hardness of B–61. The alloy was two-phase when annealed at this temperature, consisting of about 50% alpha and 50% beta solid solution. Increasing the manganese content decreased the amount of the beta phase, and decreased the strength and hardness. At 20% manganese the alloys became single-phase alpha solid solution alloys with a tensile strength of 24·5 tons per sq. in., a yield strength of 7·5 tons per sq. in., an elongation of 50%, and a Rockwell hardness of B–37. Further increase in manganese appeared to increase the strength and hardness slightly without decreasing the elongation.

The alloys were readily amenable to cold-working being readily worked by cold-rolling to at least 80% reduction in thickness. The ability of the 5% alloy to undergo this deformation in the presence of the large amount of the beta phase was noteworthy. Cold-working produced a tensile strength of 50 tons per sq. in., a yield strength of 46 tons per sq. in., an elongation of 3% and a Rockwell hardness of B–102 in the 5% manganese alloy, and a tensile strength of 47·5 tons per sq. in., a yield strength of 45 tons per sq. in., an elongation of 3%, and a Rockwell hardness of B–100, in the 20% manganese alloy.

The alloys annealed at temperatures ranging from 205° to 815° C. after 60% cold-working showed the usual changes associated with strain relief, recrystallisation and grain growth. Recrystallisation after 60% cold reduction took place between 260° and 370° C. for the 5% and 10% manganese alloys and between 370° and 425° C. for the alloys higher in manganese. The 5, 10 and 15% manganese alloys reached minimum strength and hardness with maximum elongation upon annealing at temperatures of 540° and 600° C. This corresponded to the minimum amount of the beta solid solution in their microstructure. Higher annealing temperatures produced an increase in tensile strength and hardness with a corresponding decrease in elongation because of increasing amount of the beta constituent found at these temperatures. The beta constituent was not stable under all the conditions used in this investigation. It transformed during the quenching, usually with the formation of a needlelike alpha solution structure, although it also transformed sometimes into massive alpha grains. The instability of the beta made it impossible to determine the physical properties of this phase in these alloys. When it constituted 50% or less of the structure of the alloy, beta appeared to be fairly stable and had a significant effect on the properties. The 20 and 25% alloys did not exhibit beta unless they were heated to temperatures above 700° and 750° C. respectively.

Determination of Metallic Impurities in Steel

By Georg Carlson

METHODS, which have been tested, are described for the analysis of metallic samples as well as solutions obtained by dissolving the steel in nitric or hydrochloric acid. The work is chiefly concerned with the determination of Mn, Cr, Ni, Mo, Co, V, Ti, Cu, and Sn, as well as Al in the concentration range 0·1 to 0·001%.

For the analysis of solutions, the method of evaporating to dryness a small amount of the solution on a carbon electrode has been applied. Three different light sources have been tried—the continuous direct current arc, the interrupted direct current arc, and the high-voltage alternating current arc. The interrupted direct current arc proved most suitable, and was chosen as a basis for a standard method which was further investigated and found to give a standard deviation of ± 4 to 7% for most elements.

For the analysis of metallic samples the continuous and the intermittent direct current arc were tried. The formation of molten oxide on the electrode tips was found to lower the accuracy of the analysis, and the best result, a standard deviation of ± 4 to 7% was obtained when the interrupted arc was allowed to burn only for a short interval each time it was struck, thus preventing too high a temperature at the tips of the electrodes.

The spectrochemical methods are more accurate and sensitive than the chemical methods for the determination of the smallest percentages of most elements (0·01 to 0·001%), and consequently, chemically analysed standard samples could not be used if the possibilities of the spectrographic methods were to be fully utilised. When solutions are used for the spectrographic analysis accurate standard samples may be obtained independently of chemical analysis by mixing pure solutions of the different elements, and the solution method was therefore developed and taken as a basis for other methods.

An attempt was also made to manufacture standard alloys with known composition by melting together the constituents, and these alloys were checked by the solution method with satisfactory results. Some steel samples which had been carefully analysed by chemical means, were analysed spectrographically, using metallic electrodes as well as solutions, the results showing good agreement. Fairly high amounts of alloying elements in the steel do not seem to influence the results of the trace analysis when the solution method is used, but with metallic electrodes, such an influence was found in some cases.

Most metallic elements may be determined with the described methods when present in concentrations down to 0·001 to 0·002%. The accuracy is in most cases ± 0·001% for amounts less than 0.020% and ± 0·005%, for amounts of about 0·1%. The solution method may be used with advantage in microanalysis. Quantitative determinations may be carried out with a total sample weight of a few hundredths of a gram.

From *Jernk. Annal.*, 129, **5**, 1945.